Agenda
Help
Summary

HIGH RATE E-BEAM YBCO PROCESS ON TAPE

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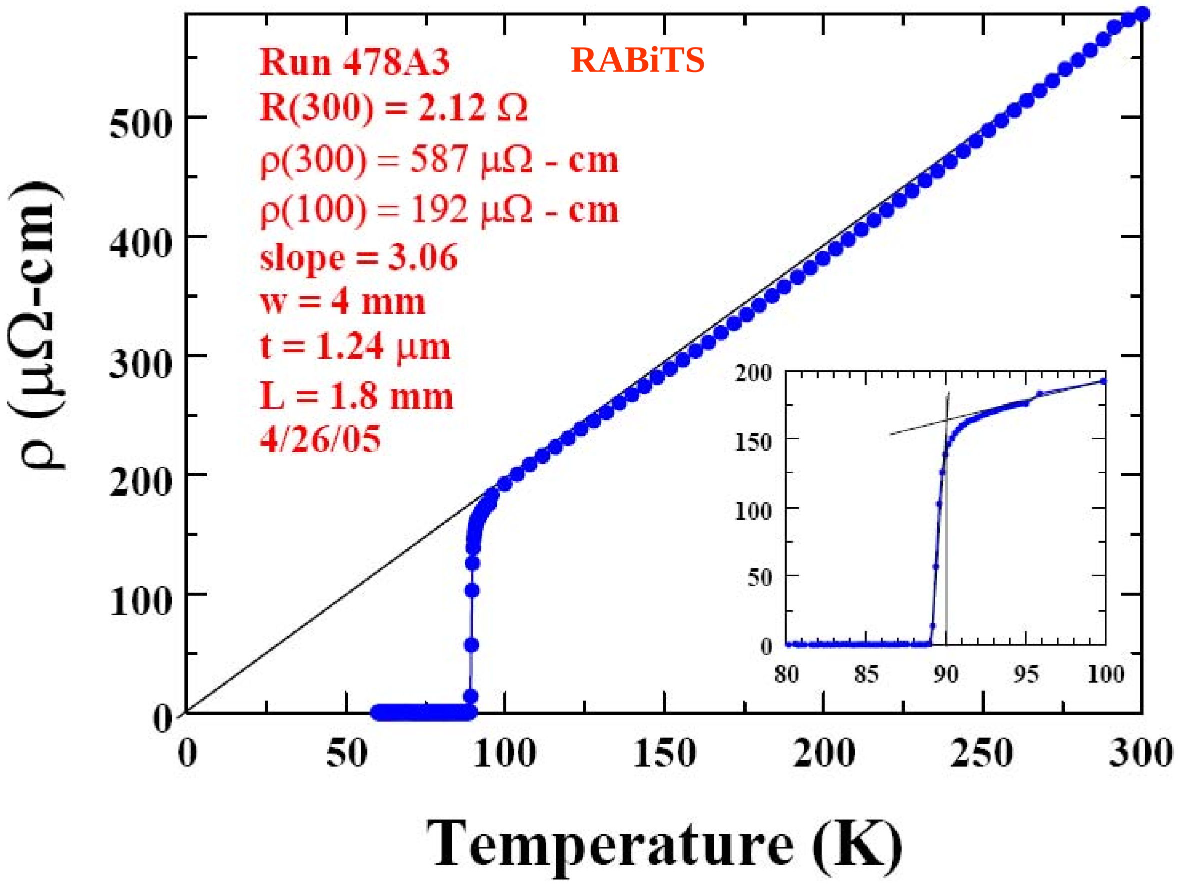
***AFOSR**
**** DOE support:$ 36k**

- Introduction - Storer
- Materials science – Stanford - *Hammond*
- Reel-to-Reel processing – LANL - *Storer*
- Compulsory slides - *Storer*
- Questions – *Storer/Hammond*

Co-evaporation produces high Jc films

1.0 MA/cm^2 (SF, 77K) 1.1 micron thickness



Evaporation has high throughput potential for making thick films

Multi-hundred kW systems evaporating kg/hour exist in industry.

-

Examples:

Turbine blades

Titanium Metal Matrix Composites

Most likely: least expensive raw materials

Results at Stanford show that liquid facilitated growth results in rapid growth of YBCO.

- High J_c > 1 MA/cm^2
- Lower fall-off with thickness -> higher I_c
- Thick film potential > 5 microns
- Wide area at high linear speed (throughput)
- Pure, least expensive feedstock

Outline

- Goals: (AFOSR Sponsorship)
  - Phase stability of YBCO
  - Phase equilibrium of thin films, role of liquid Ba-Cu-O
  - Growth morphology
- Introduction
  - Tools
    - Evaporation facility
    - FTIR
    - XRD – Dome
- Example of FTIR
  - Determination of liquid $BaCu_2O_3 \rightarrow (s)CuO + (s)BaCuO_3$ in pressure, temperature
  - Monitor YBCO formation

Outline

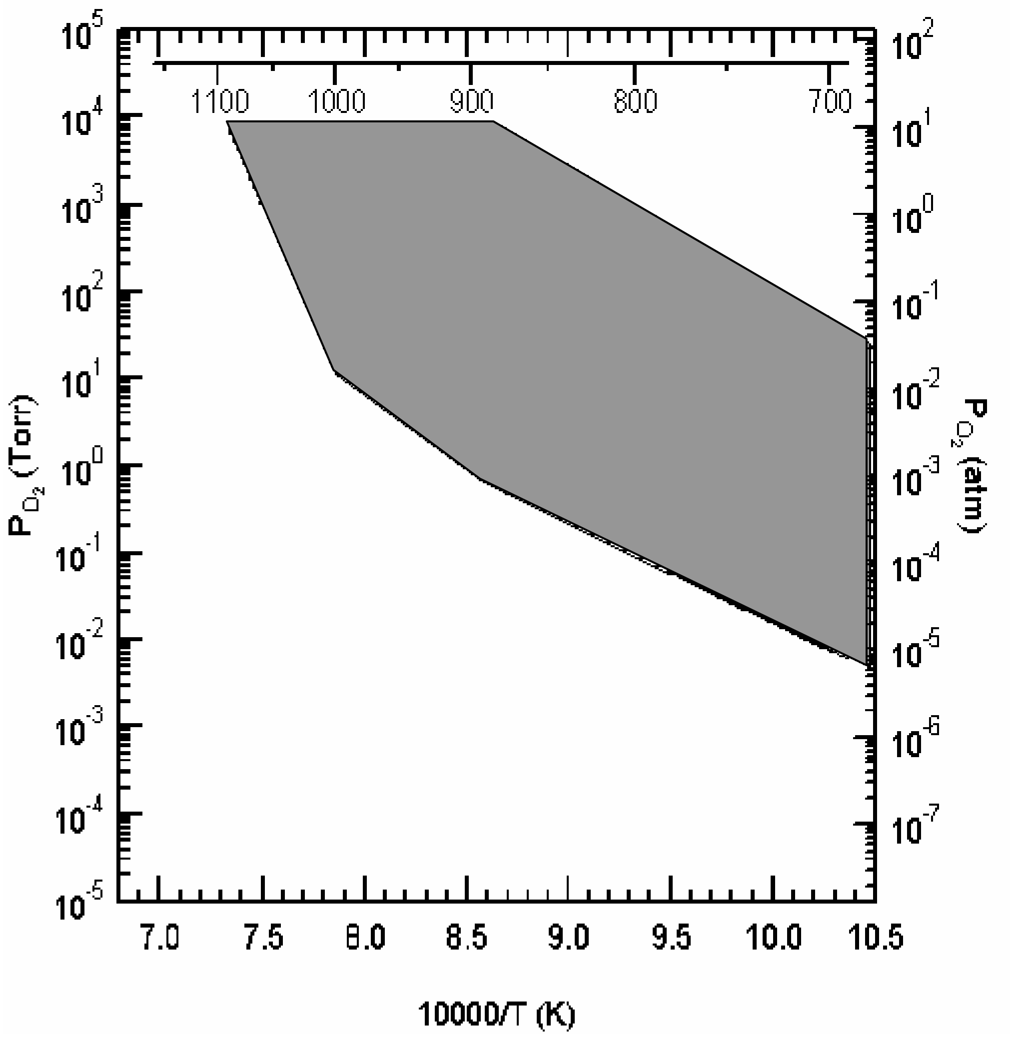
- Example of Dome - XRD
  - Growth of precursor YBCO at 830°C as add O_2
    - Reaction of CeO_2 and YBCO
    - Comparison of growth at three O_2 pressures
    - Growth of low temperature precursor YBCO as function of temperature at three fixed O_2 pressures

- Lessons learned in XRD – Dome

- Processing in evaporation chamber
  - In-situ with pause
  - Data: Jc ~ $1MA/cm^2$, XRD, R(T) on RABiTS
    Jc(H,77) – compared with PLD on RABiTS
  - TEM – Lateral growth

- Substrate used: LAO crystals, RABiTS(AMSC), IBAD-MgO(LANL)

YBCO stability diagram



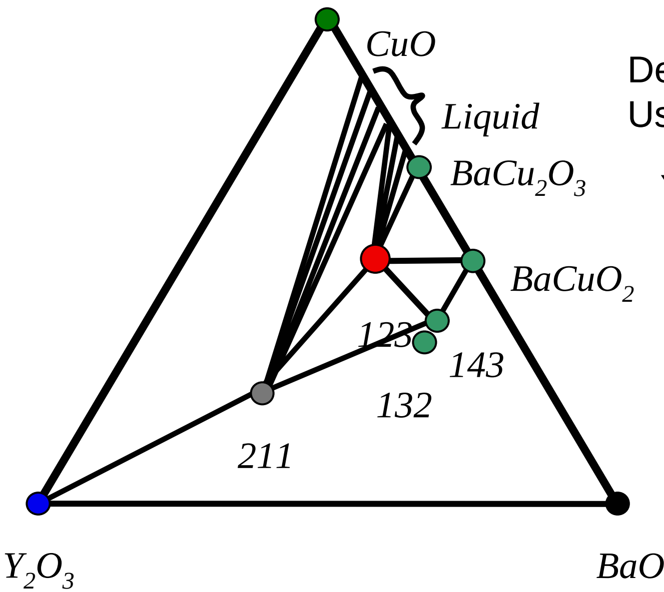
region where YBCO is stable (P,T)

where liquid Ba-Cu-O transition;

(L)$BaCu_2O_3 \rightarrow$(S)CuO+(S)$BaCuO_3$

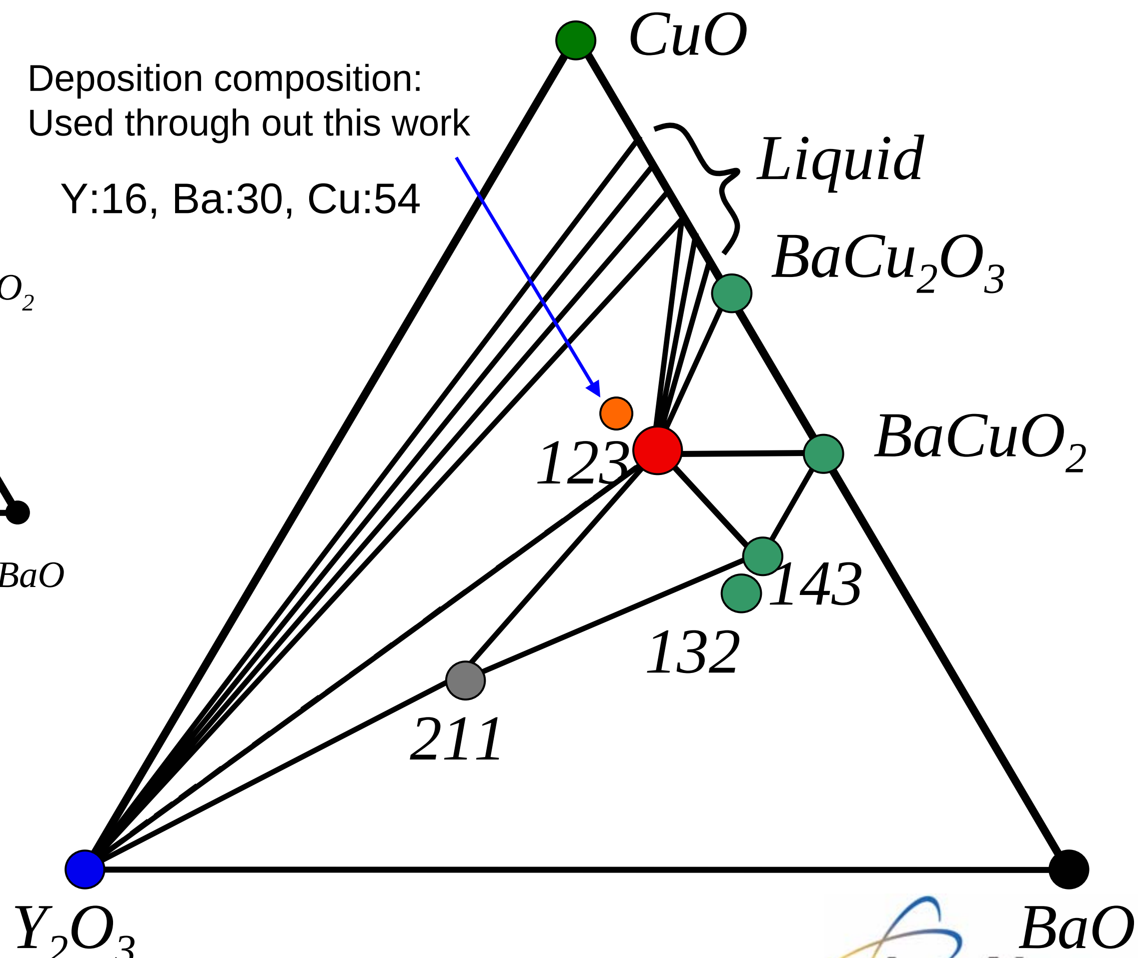
Phase Diagram

Expected phase diagram
(Wong-Ng, Cook)



T > T$_{MELT}$ Ba-Cu-O

Proposed metastable Diagram
Based on Y_2O_3 - BaCuOx-123



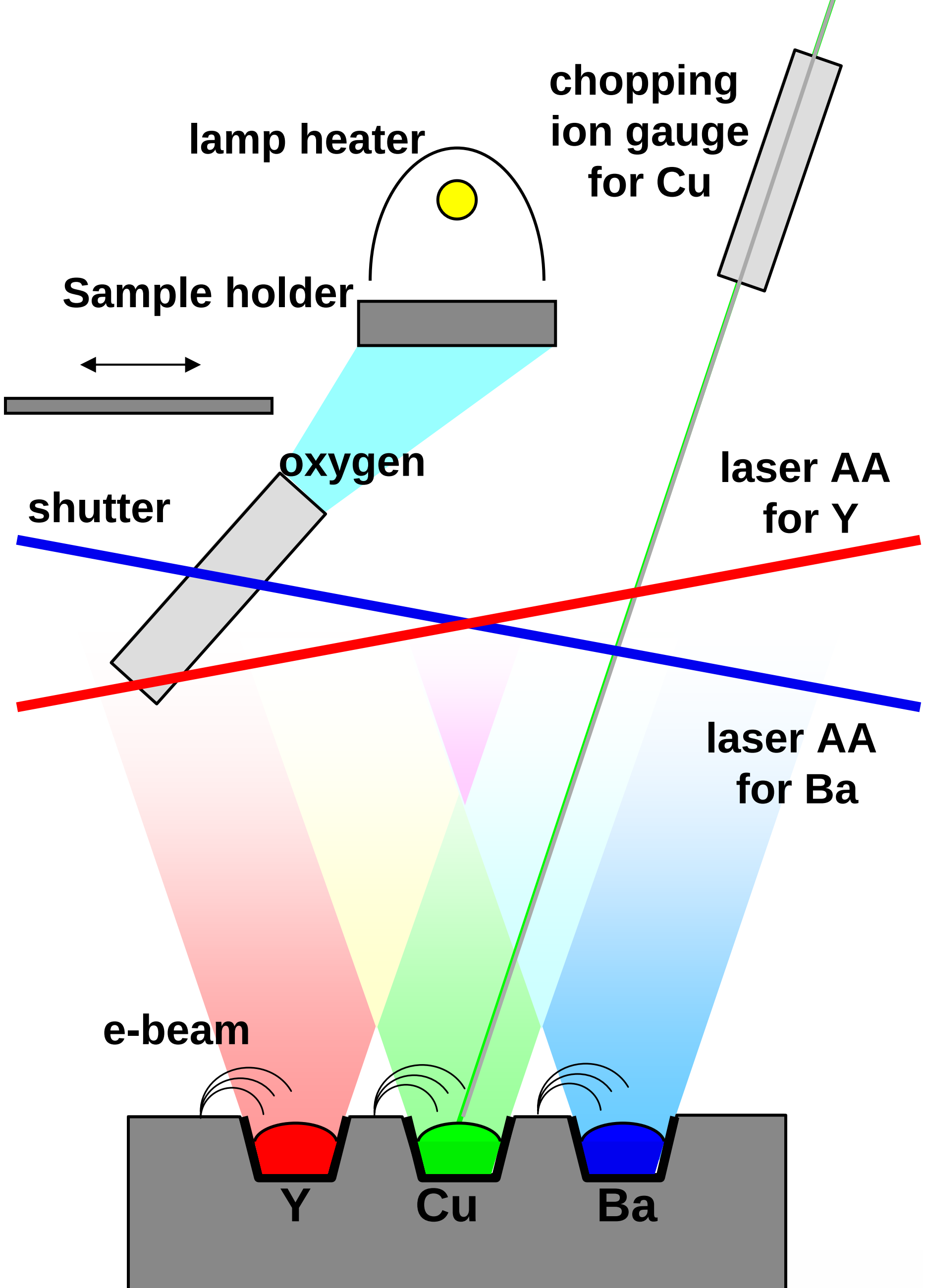
Film growth method

Electron-beam reactive co-evaporation

Composition control
- laser AA: Y & Ba
- chopping ion gauge: Cu

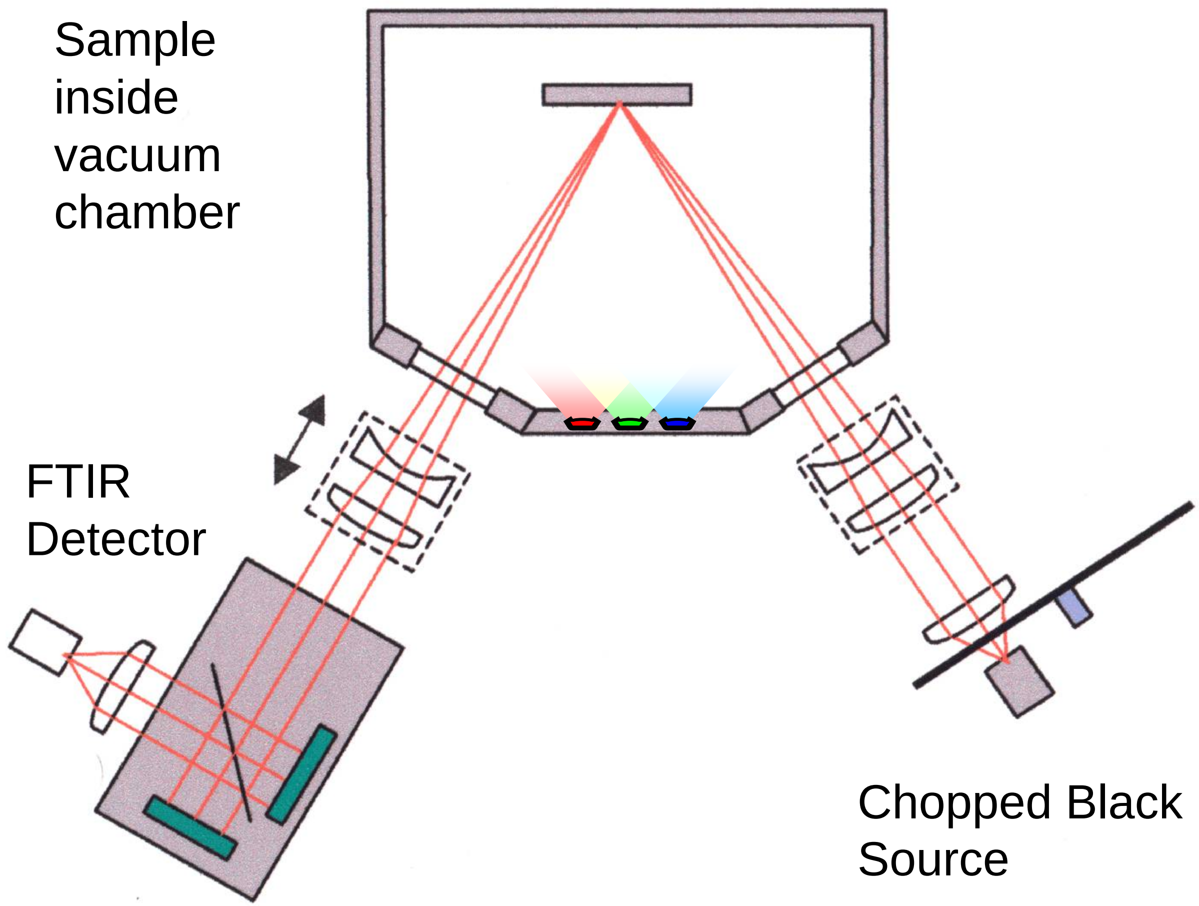
Deposition rate
100 ~ 300 Å/s

Deposition pressure
5×10^{-5} Torr



Fourier Transform InfraRed set up

Used to monitor dielectric properties during and post deposition wave lengths 500 to 6000cm^{-1}



XRD Dome Experiments

Controlled Atmosphere XRD

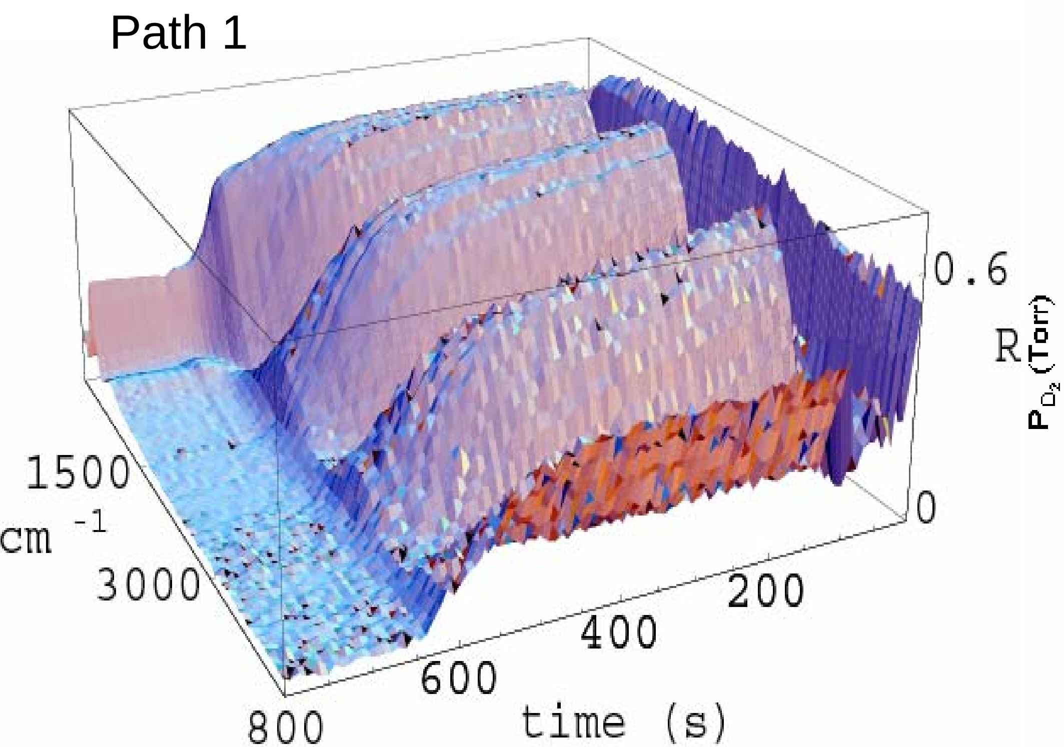
XRD heating dome

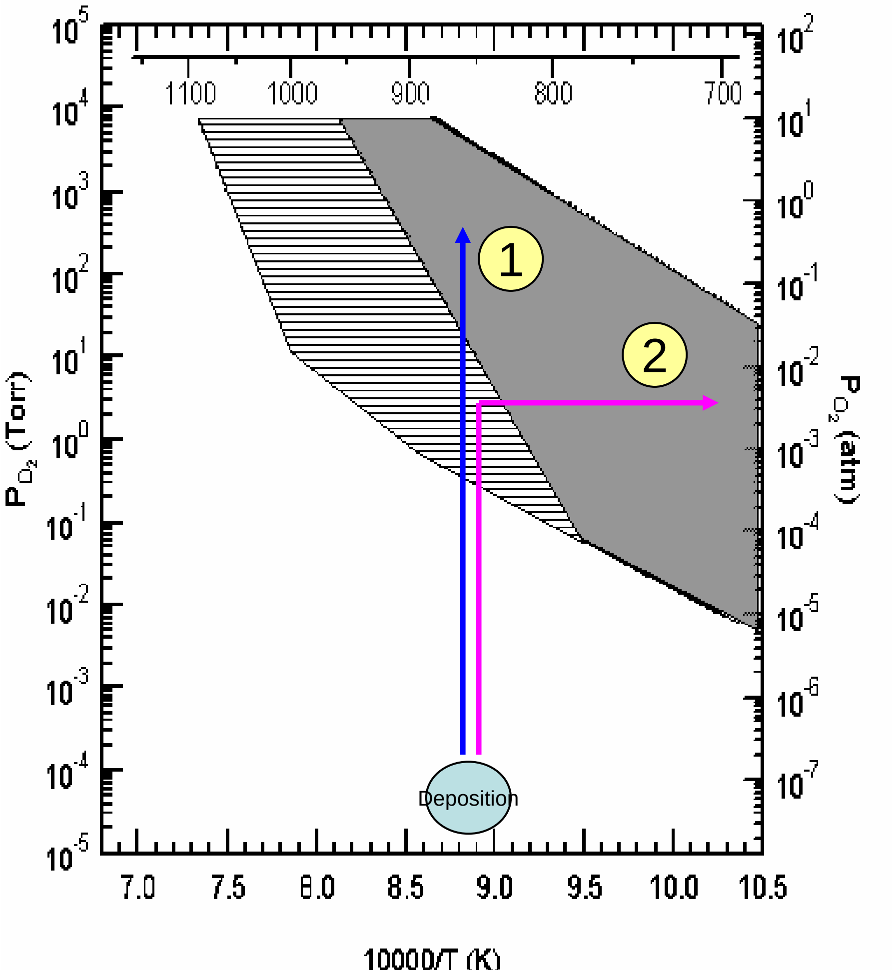
Capable of taking XRD measurements while heating the sample under a certain environment

Great way to find the post-deposition Condition for YBCO

Example of FTIR

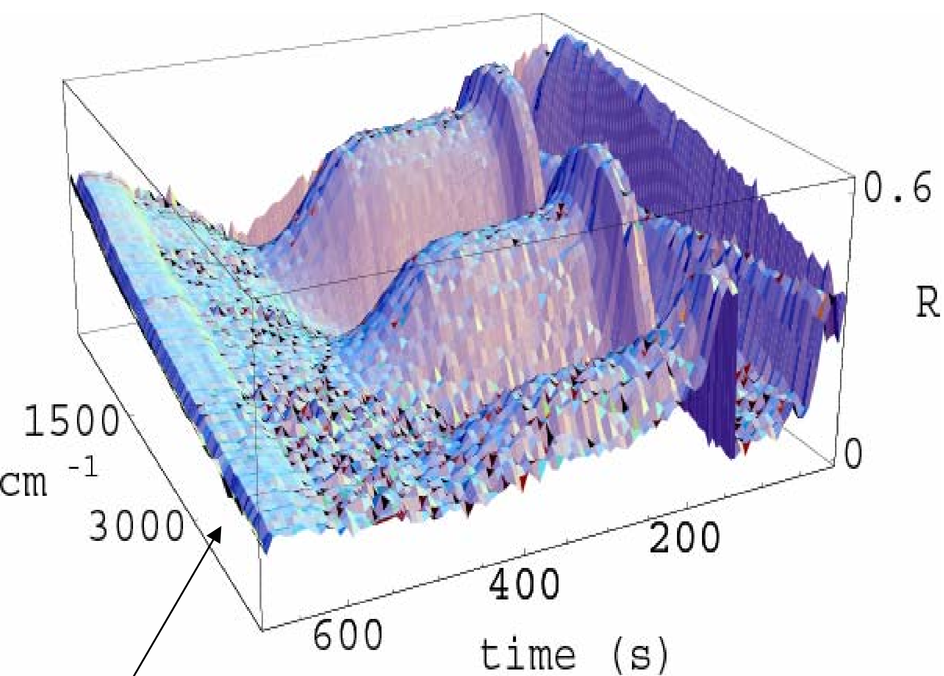
Determination of liquid Ba-Cu-O decomposition: $BaCu_2O_3 \rightarrow (s)CuO + (s)BaCuO_3$



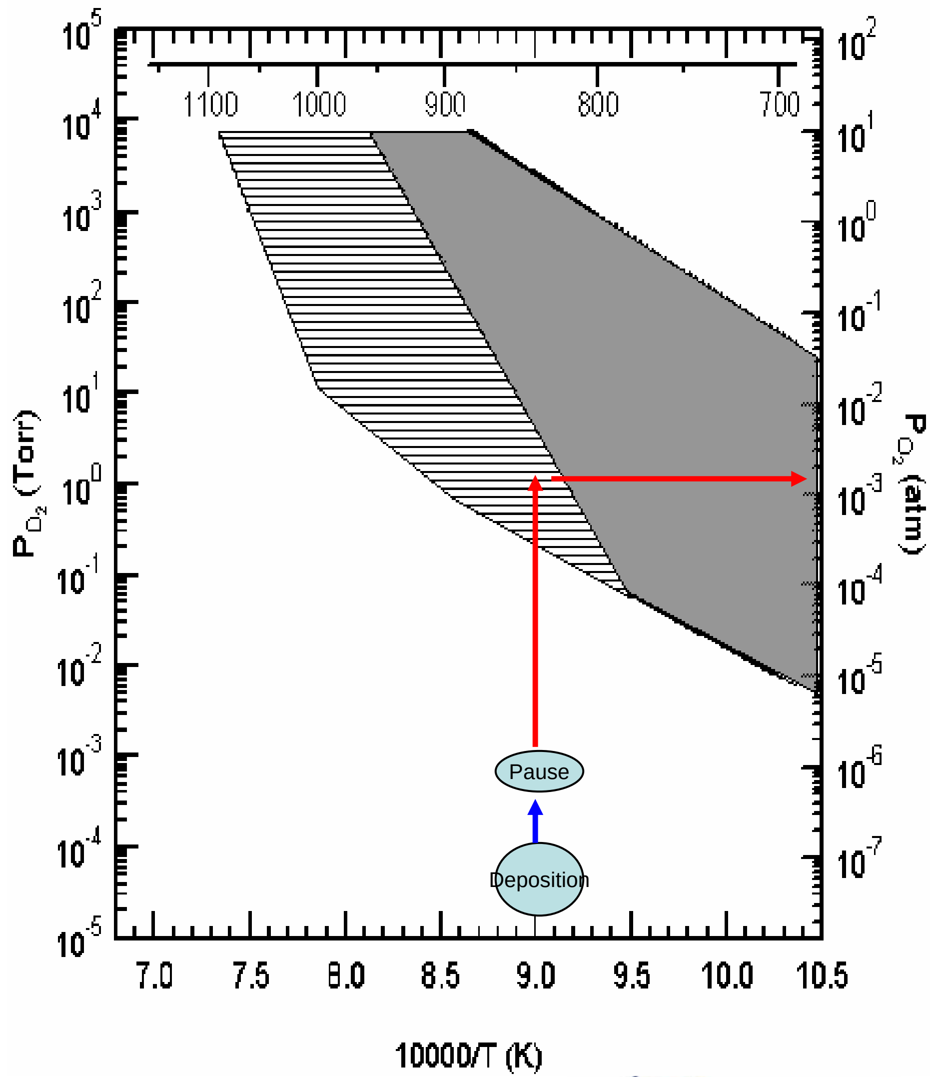


Path 2 - No change till ~ 650°C: supercool liquid → Glass

FTIR during and After YBCO Deposition



Drude metallic
YBCO

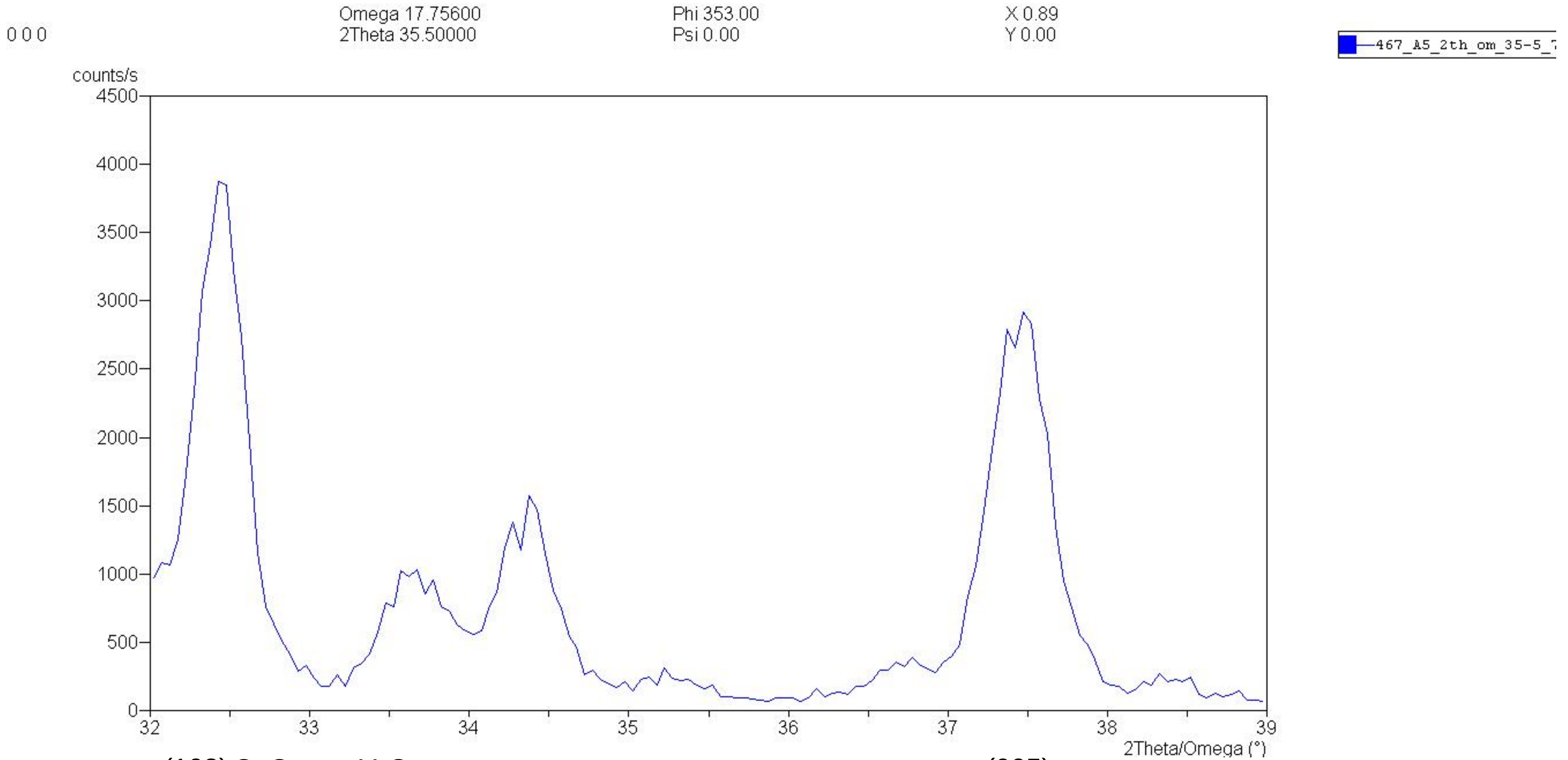


Examples of XRD Taken in Dome

A. Time sequence of growth on RABiTS: /CeO_2/YSZ/Y_2O_3/Ni(W)

Reaction: YBCO + $CeO_2 \rightarrow BaCeO_3$ – Does this prevent Epitaxial growth?

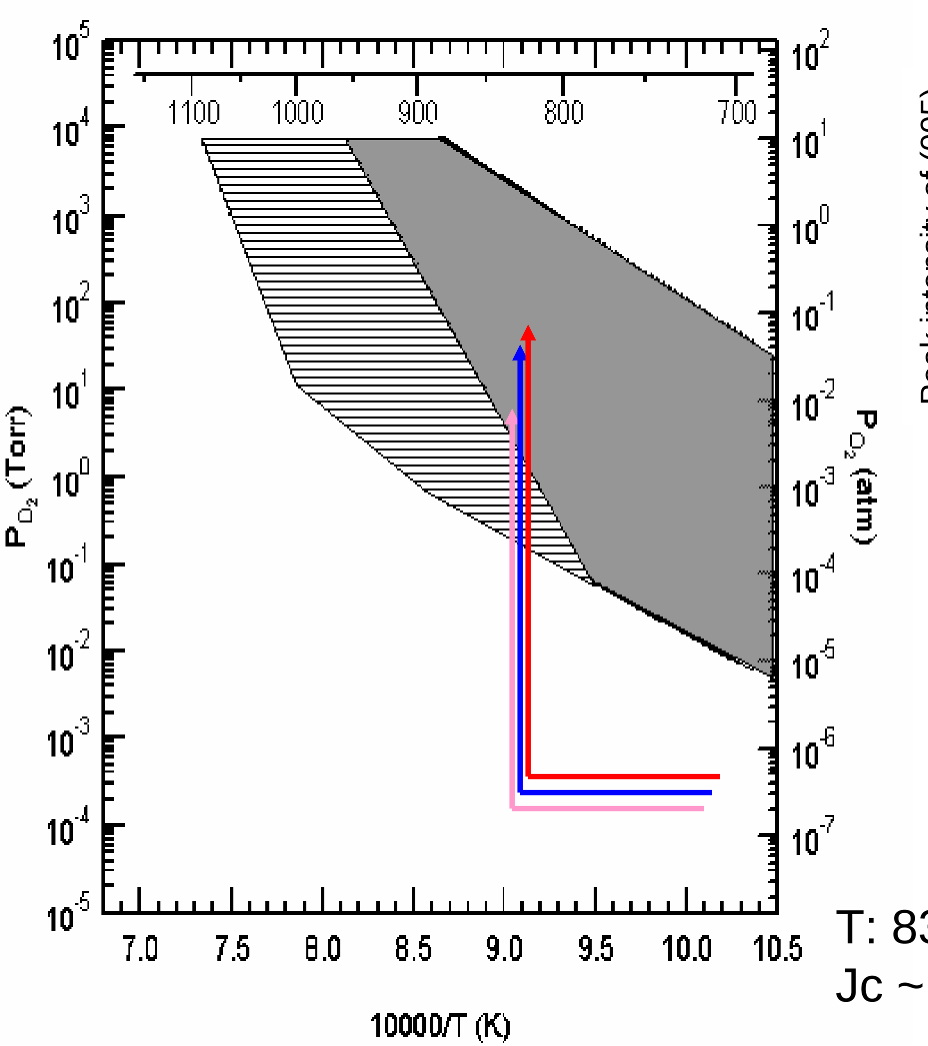
Monitor YBCO (005) & CeO_2



(103) CeO_2 Y_2O_3 YSZ (005)

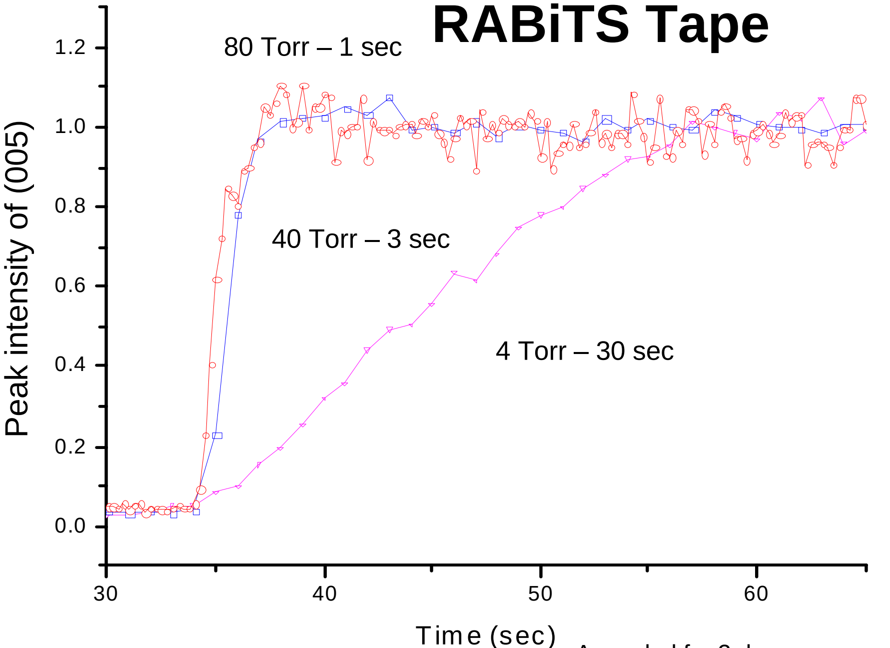
Conclusion: 123 has nucleated C-axis before CeO_2 decays

Example of XRD Dome:
B. Traces of temperature and pressure with the dome experiment:

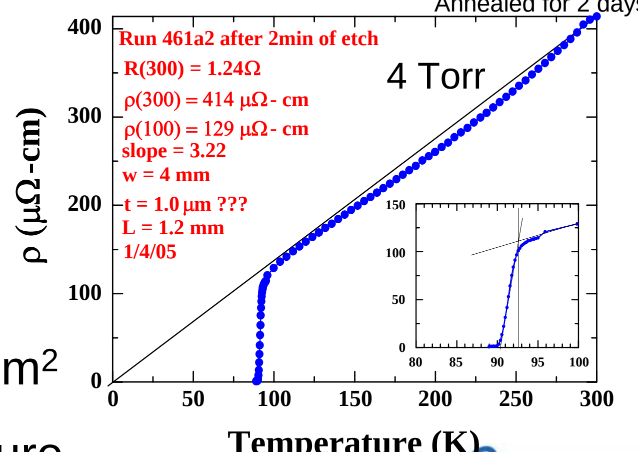


T: 830˚C
Jc ~ 2kA/cm²

RABiTS Tape



Annealed for 2 days

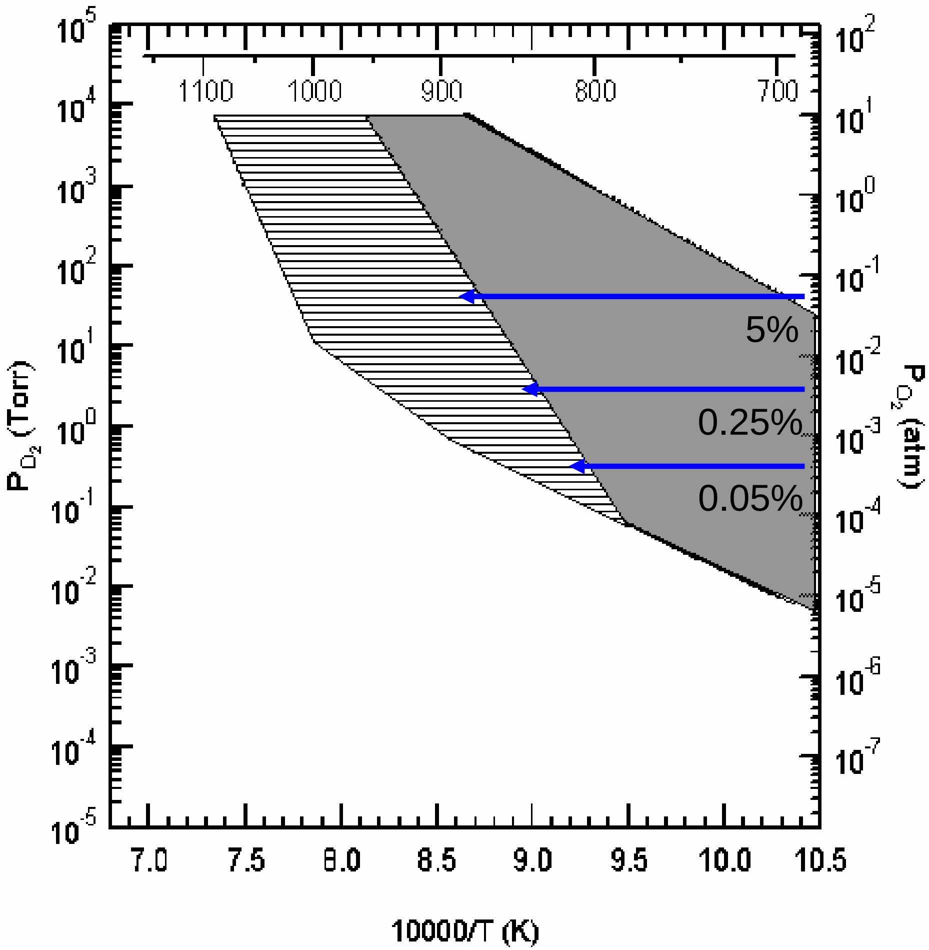


Growth occurs within seconds at higher pressure

Example of XRD Taken in Dome:

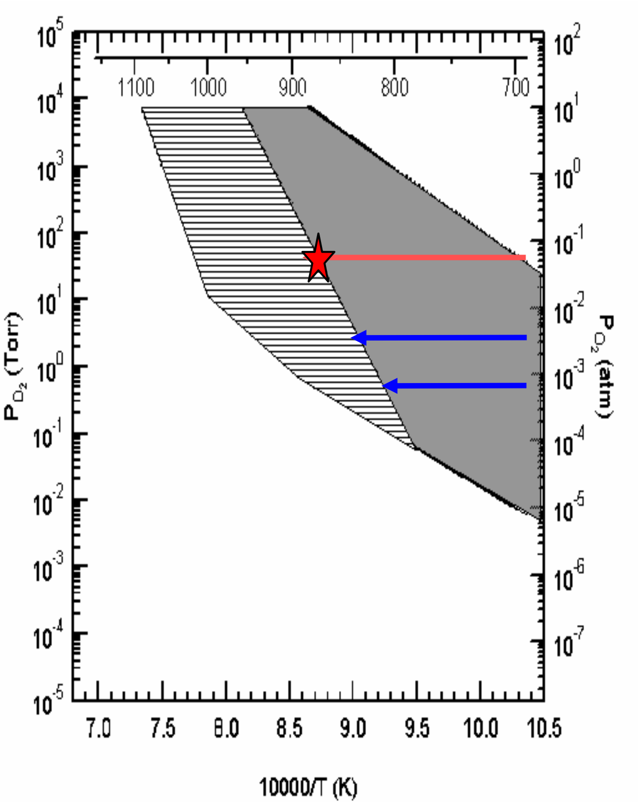
C. Growth of low temperature precursor

Investigate where in Po_2, T growth occurs

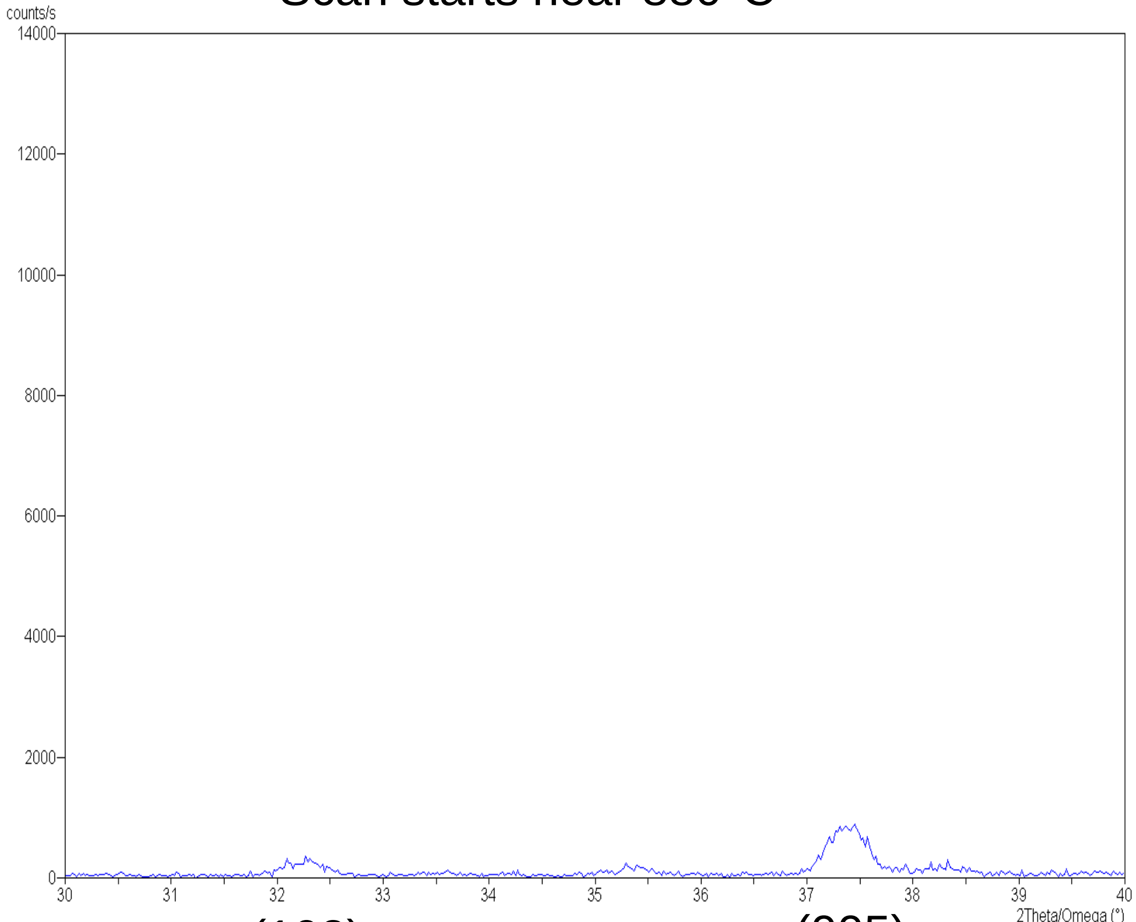


- Samples made at 300°C in 5×10^{-5}Torr O_2 on LAO
- Mount in dome heat to 650°C in Argon
- Heat in three Po_2

Heating in 5% Po_2

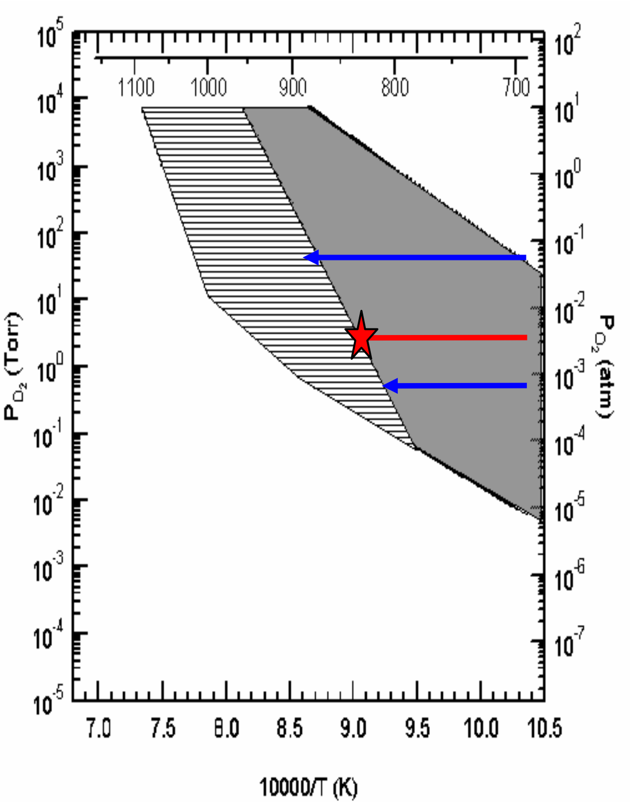


Scan starts near 880°C

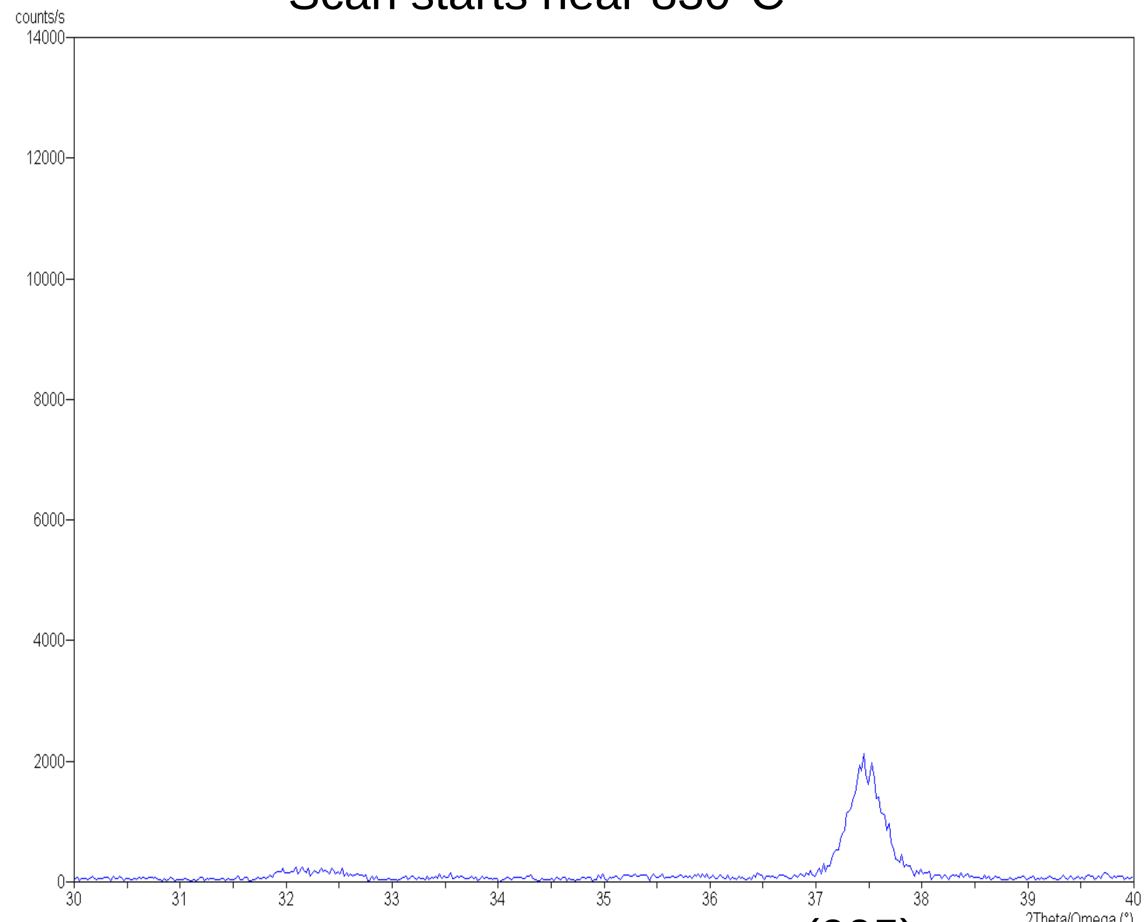


(103) (005)

Heating in 0.25% Po_2

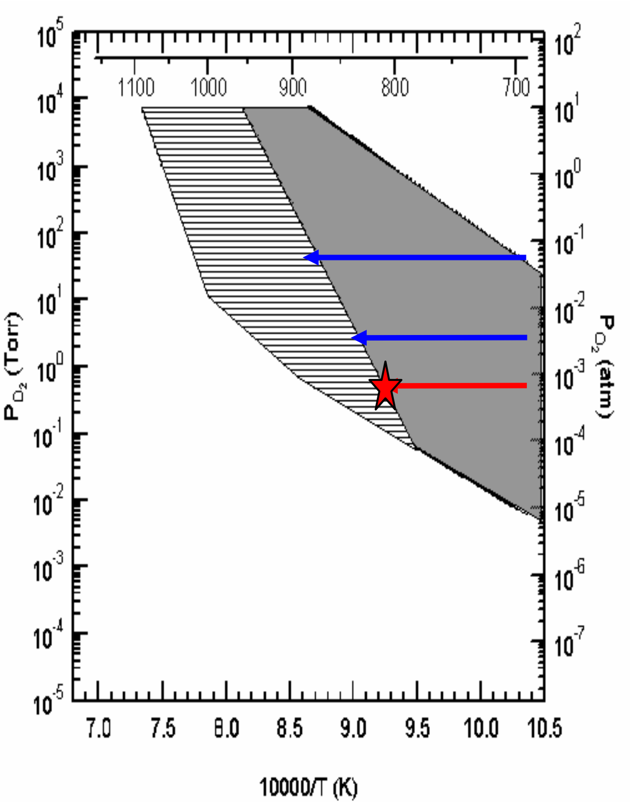


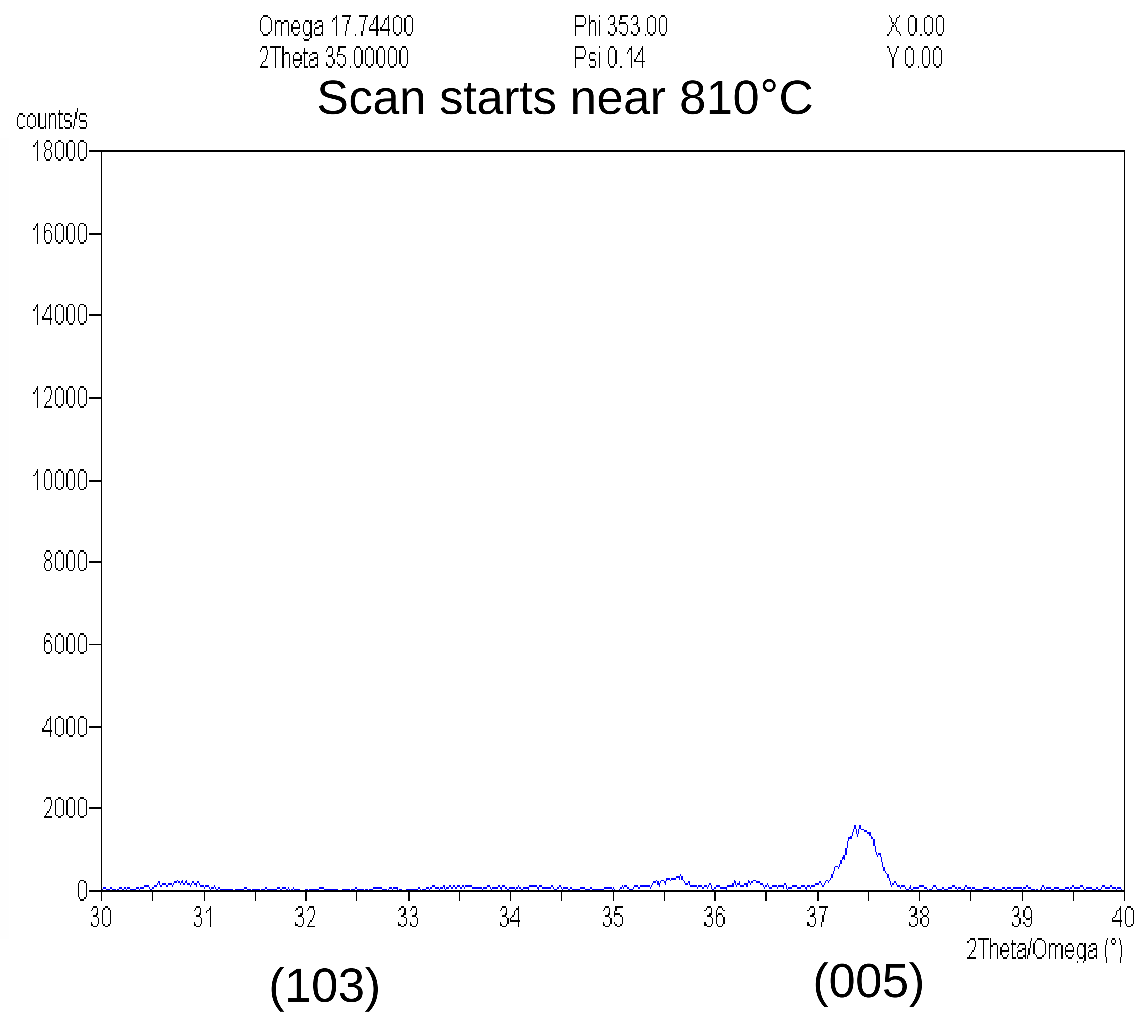
Scan starts near 830°C



(103) (005)

Heating in 0.05% Po_2





Summary

Sample #	% O_2	Rapid Growth Temp	Time for Growth	(005)/(103)
489A1	5	880°C	2 min	13
489A6	0.25	830°C	5 min	25
492A1	0.05	810°C	8 min	110

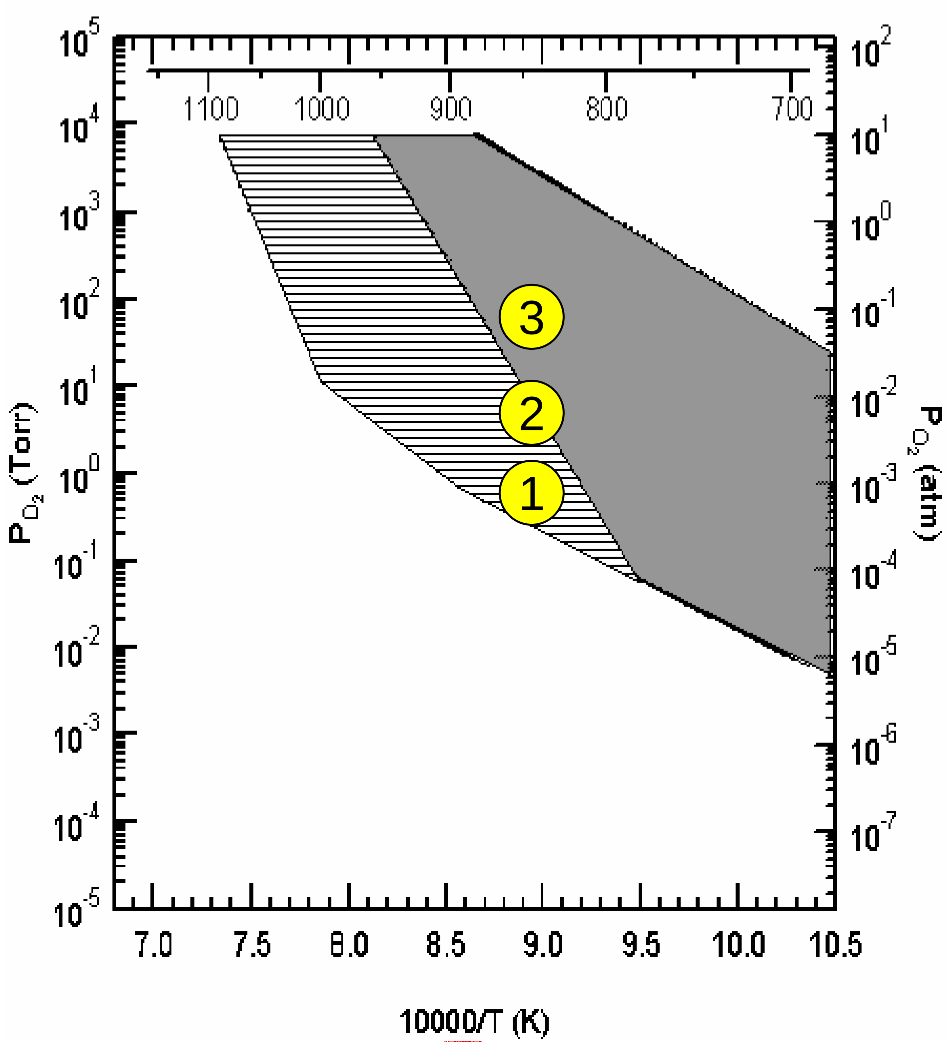
On LAO

Conclusions:

- Majority of growth occurs near liquid line
- Rate of growth higher PO_2, higher temperature
- (103) less at low growth rate, low temperature

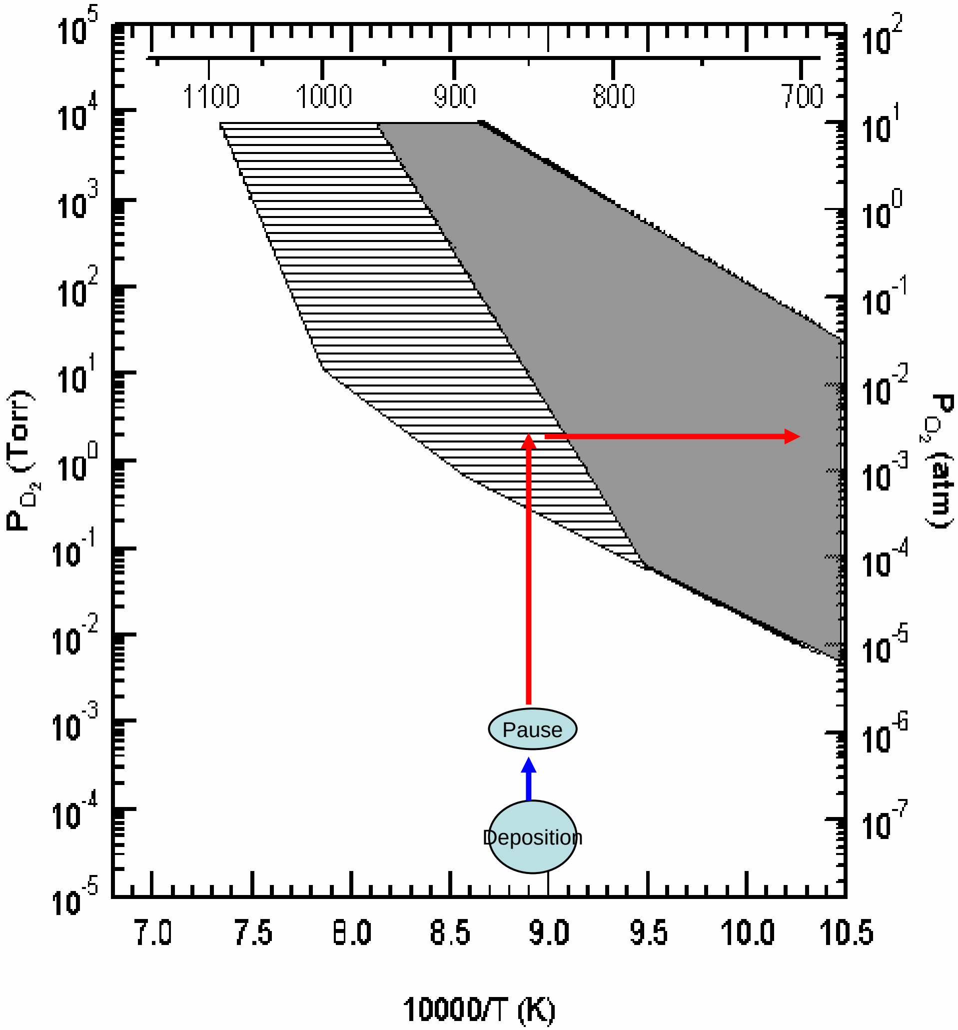
Lesson Learned in XRD Dome

RABiTS and IBAD-MgO-LMO

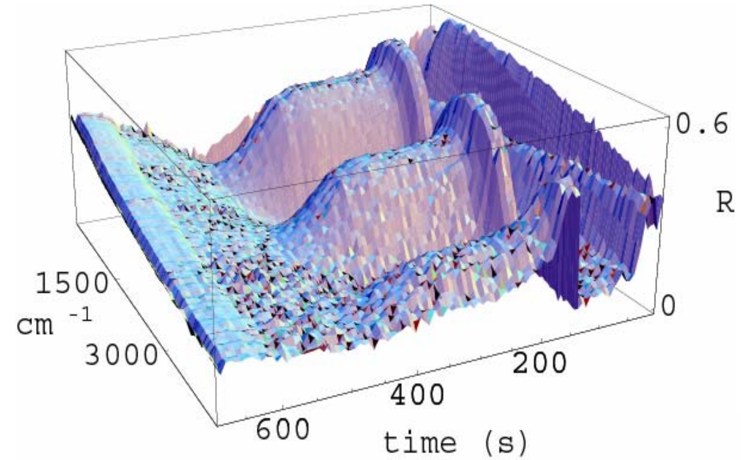


- Region 1
  - Slow growth
  - “Perfect” c-axis
  - Hard to get O_2 in
  - No pinning << 1MA/cm^2
- Region 2
  - Fast growth
  - New tech required to suppress (103) → Jc ~ 1MA/cm^2 (1.1μm)
- Region 3
  - Polycrystalline
  - Homogeneous nucleation
  - Very rapid growth < 1sec

Processing in Evaporation Chamber

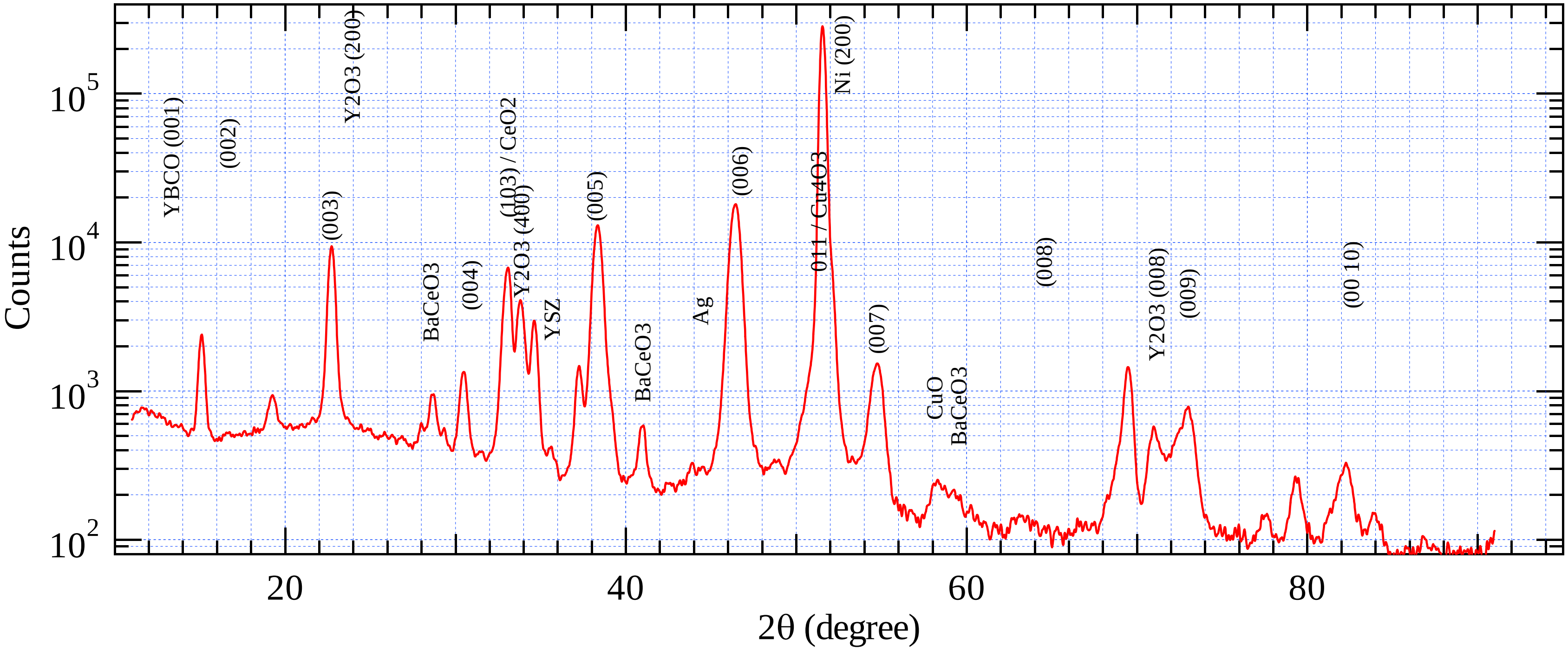


- Deposit at 5×10^{-5}Torr O_2, 830°C
- Add O_2 2mTorr and pause for seconds
  - Form "Glassy" state
- Add more O_2
- Cool down in O_2
- 300°C, Atmosphere O_2



Processing in Evaporation Chamber
(cont)

Sample 478A3 (1.1μm) – RABiTS (AMSC)



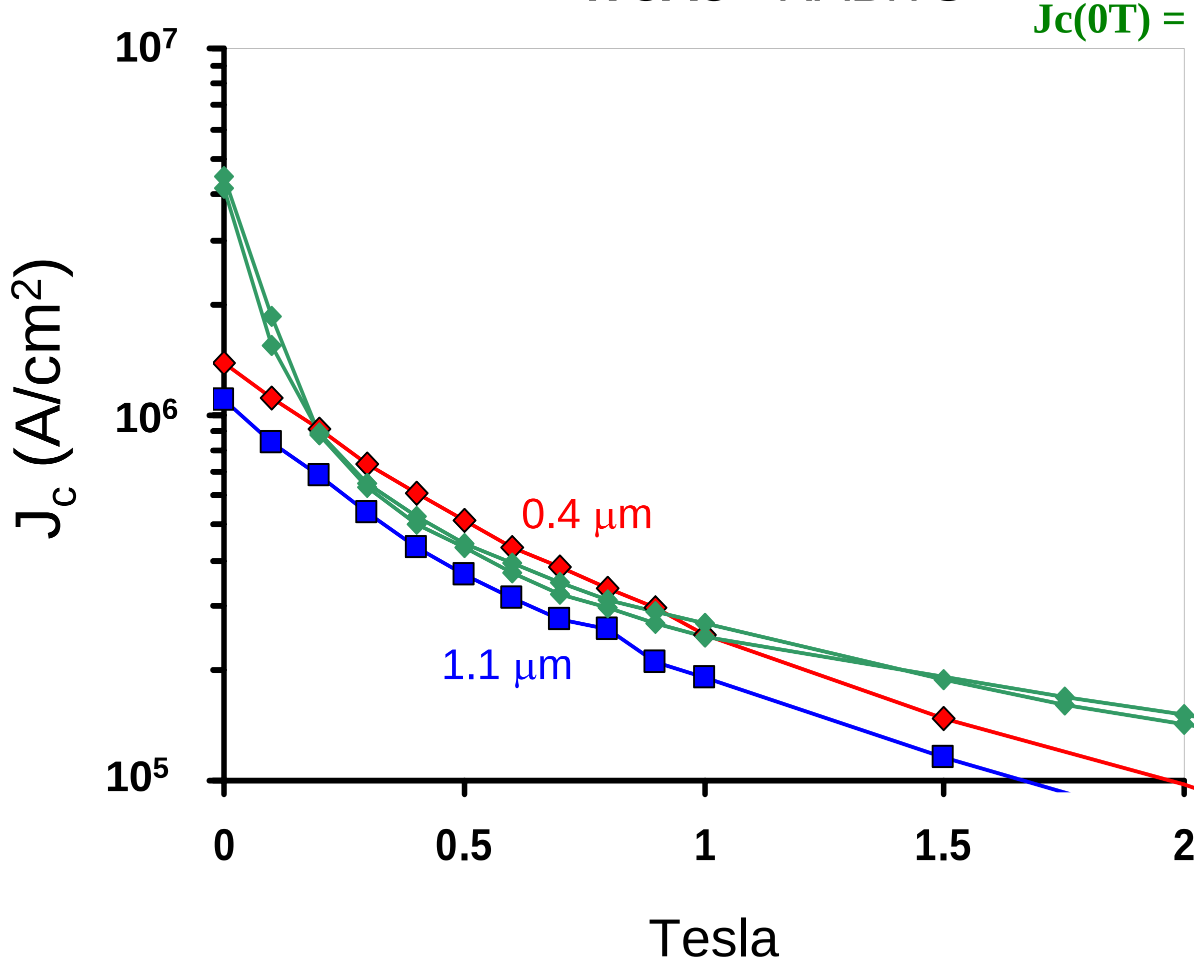
Note: Small(103), No a-axis, $BaCeO_3$ formed by reaction

Feldmann: Measured and Compared

Jc: 1.1um
0.4um – thinned } Stanford
PLD/RABiTS - ORNL

0.6 micron PLD/RABiTS
Cantoni et al (ORNL)
(intra-grain/single crystal)
Jc(0T) = 4.2,4.5 MA/cm2

478A3 - RABiTS



Feldmann: Measured and Compared

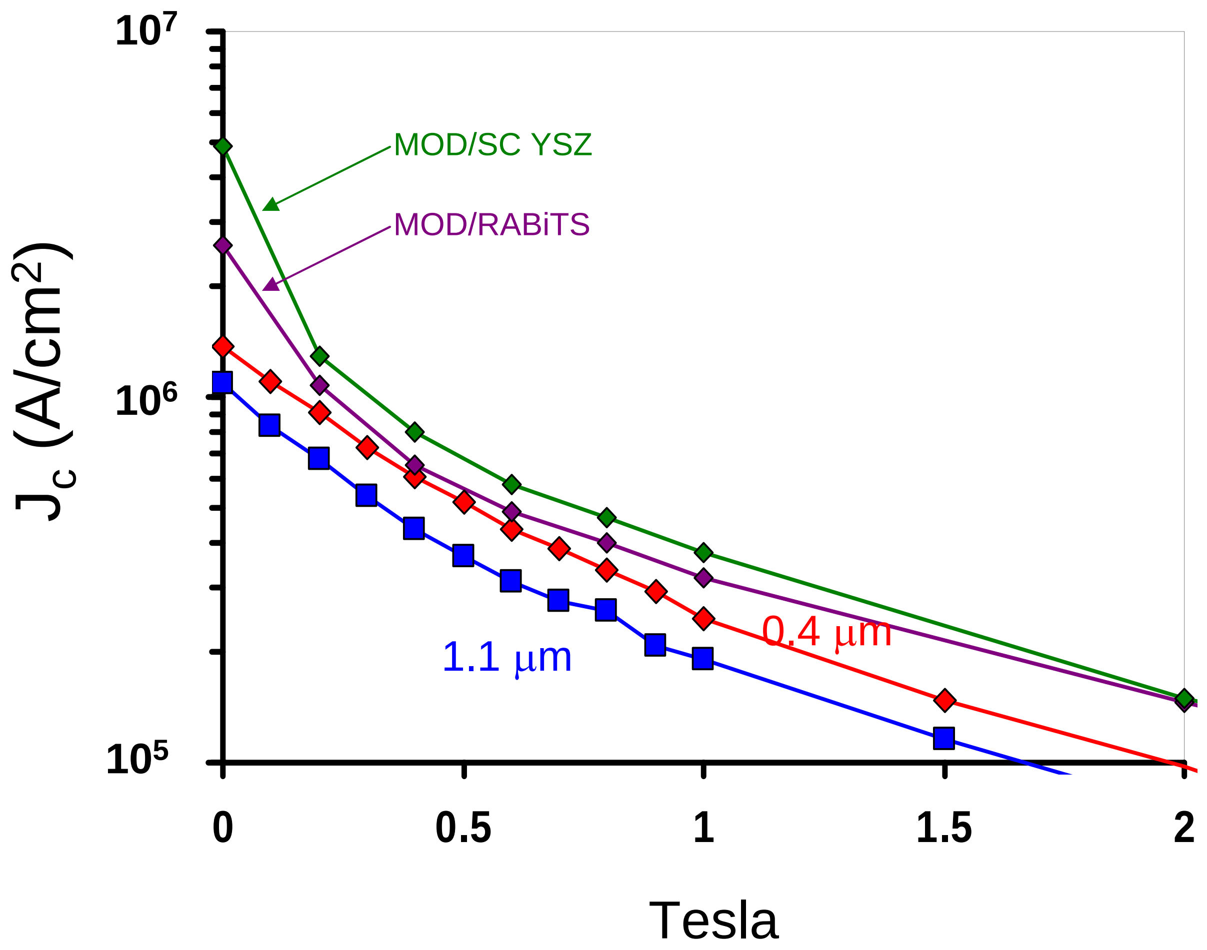
Jc: 1.1um

0.4um – thinned } Stanford

MOD – Kim et al (AMSC)

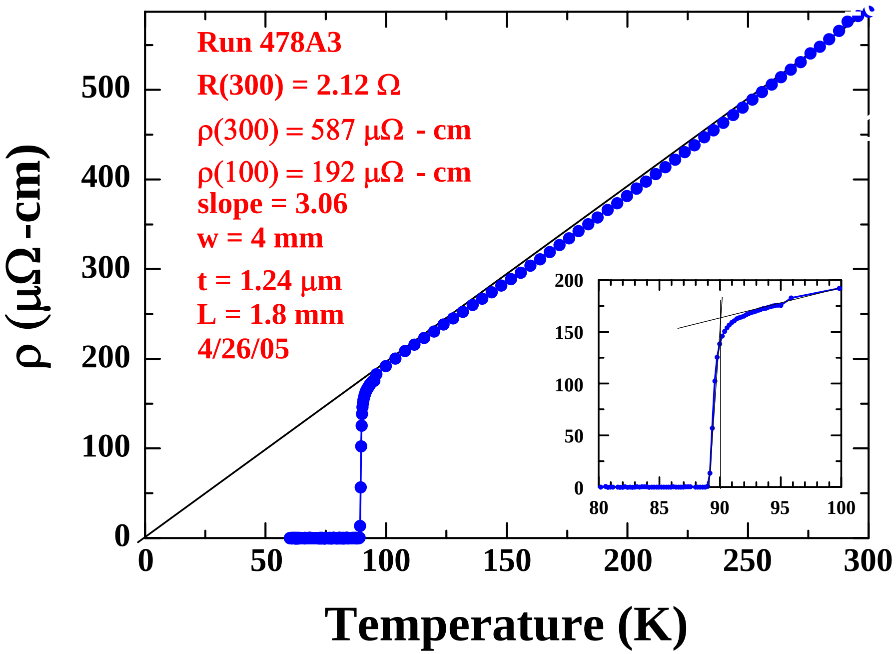
MOD data from:
Kim et al, PRB **71**, 104501 (2005)

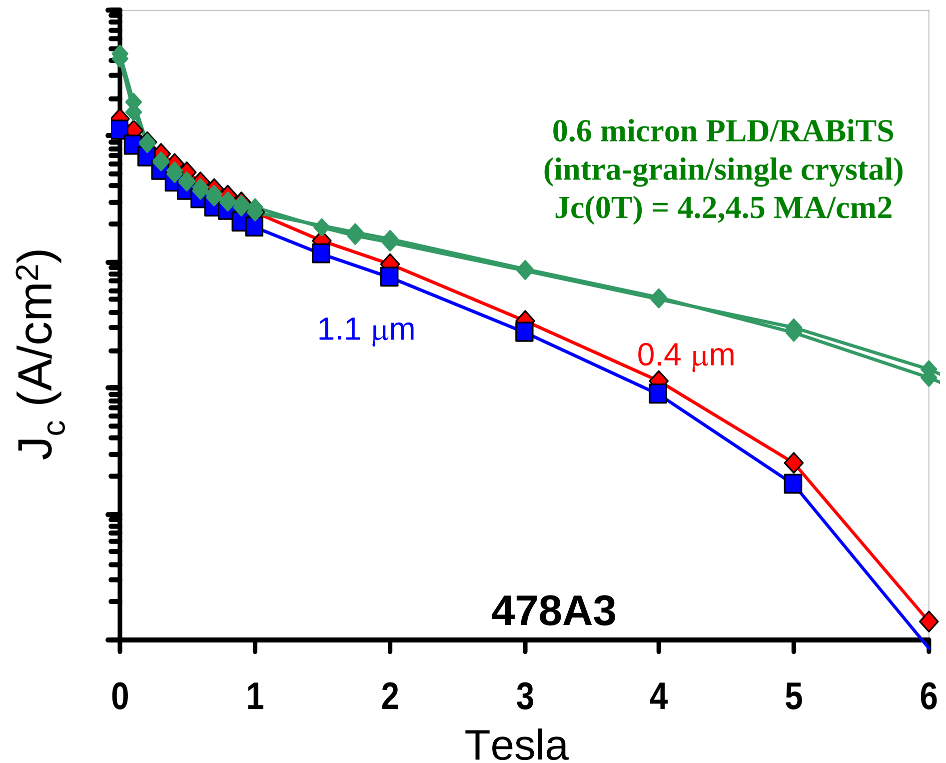
478A3 - RABiTS



Processing in Evaporation Chamber
(cont)

Jc(H): Feldmann-Wisc.
Thinned 1.1 → 0.4um, Slight change





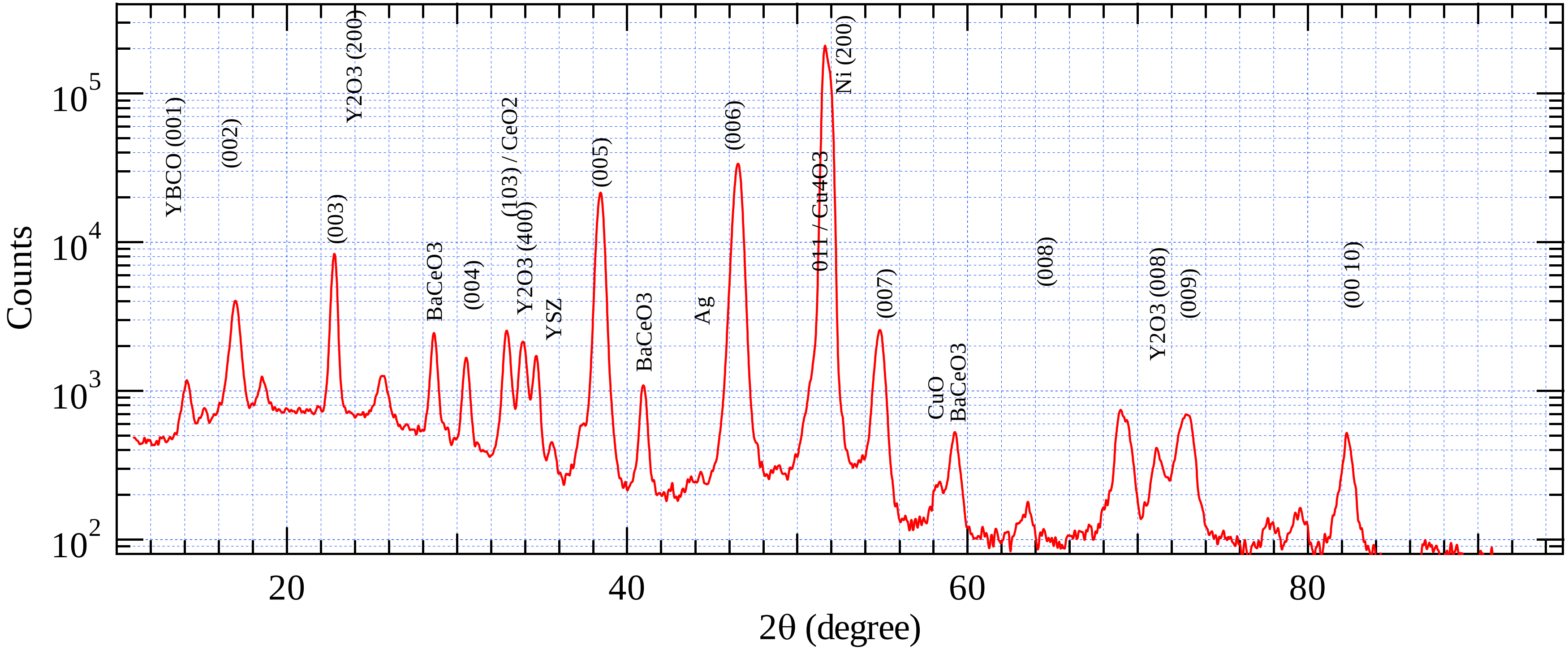
Note: No thickness dependence 1.1 – 0.4µm

Suggests laminar growth grain structure, liquid phase involved

Comparable with 0.6µm PLD with Jc(0) = 4.2 MA/cm2

Processing in Evaporation Chamber
(cont)

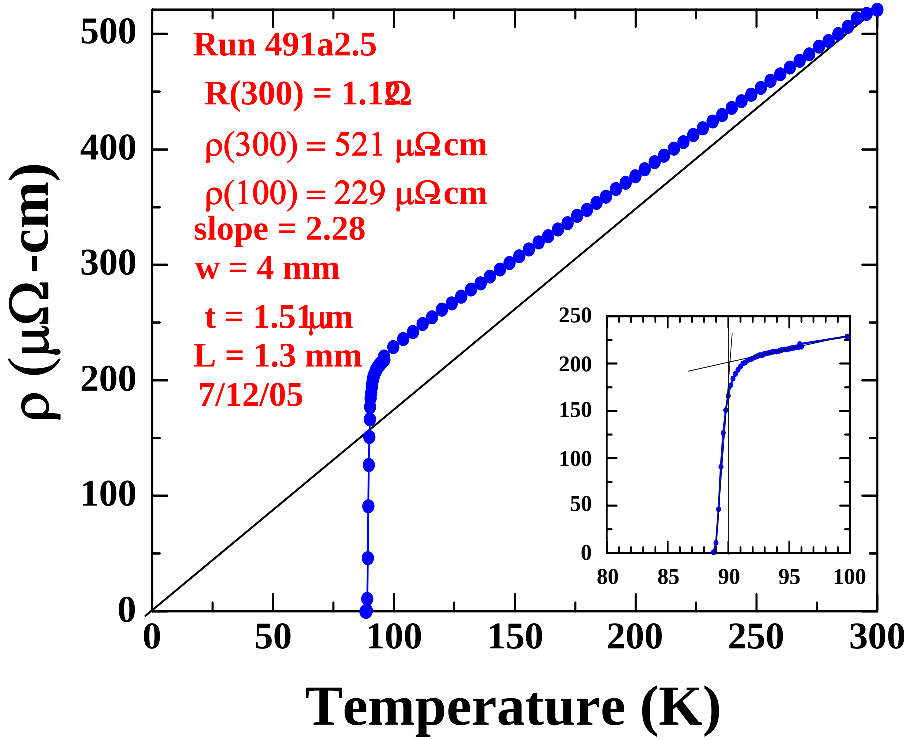
Sample 491A2.5 - RABiTS

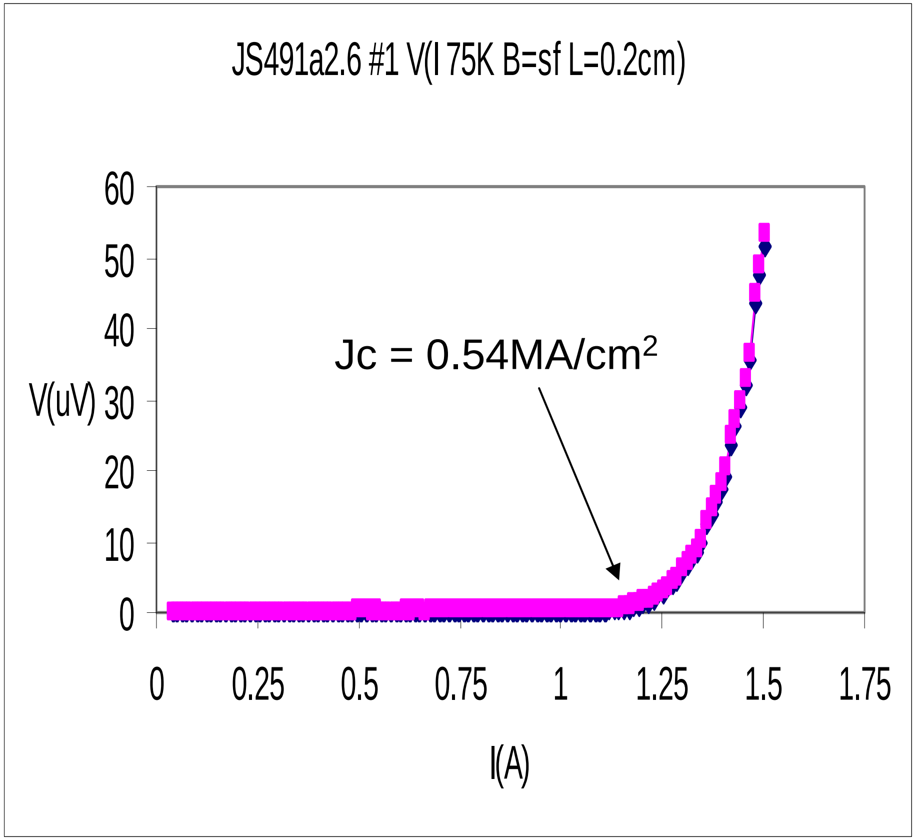


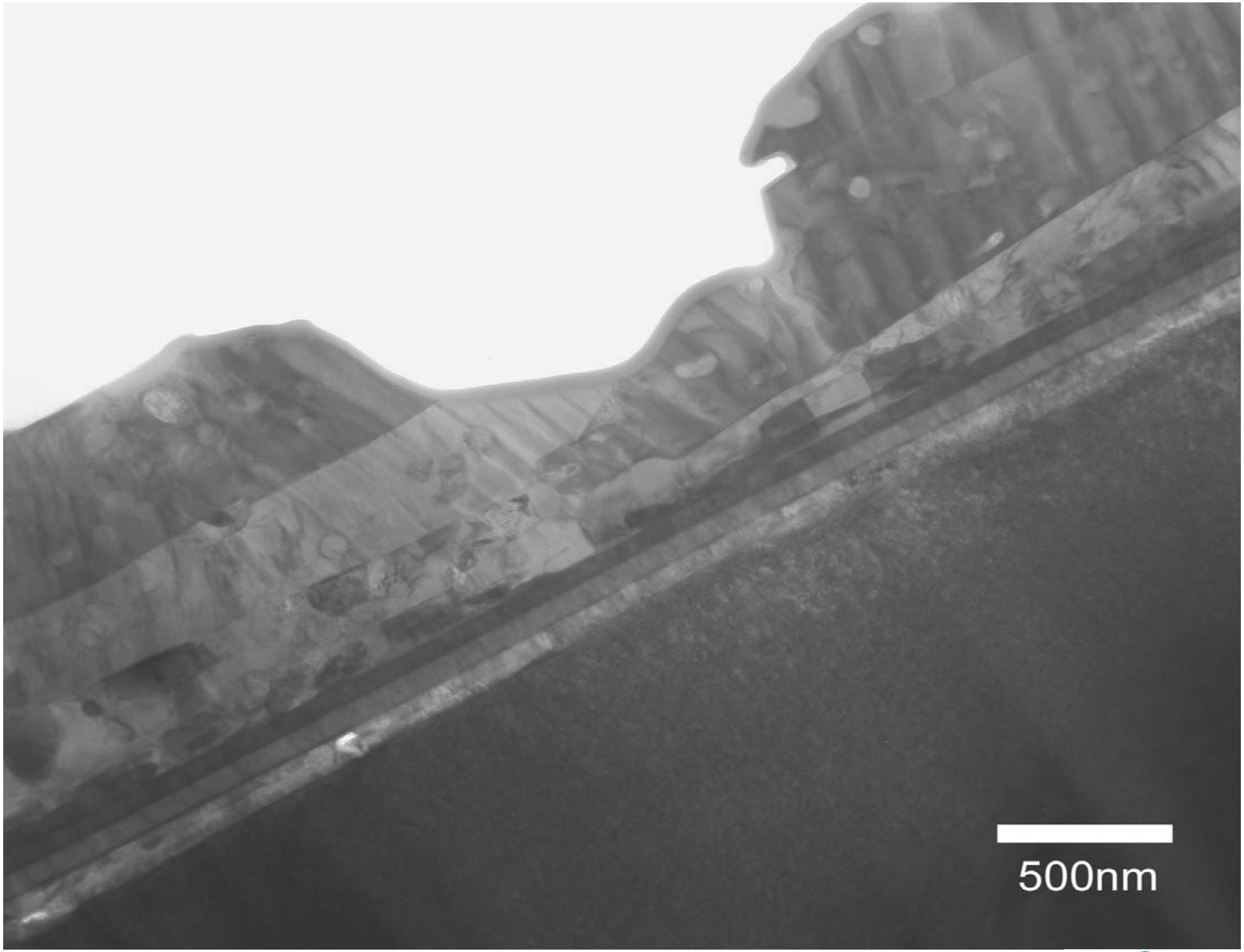
Very good clean C-axis YBCO

Processing in Evaporation Chamber
(cont)

491A2.5 - RABiTS

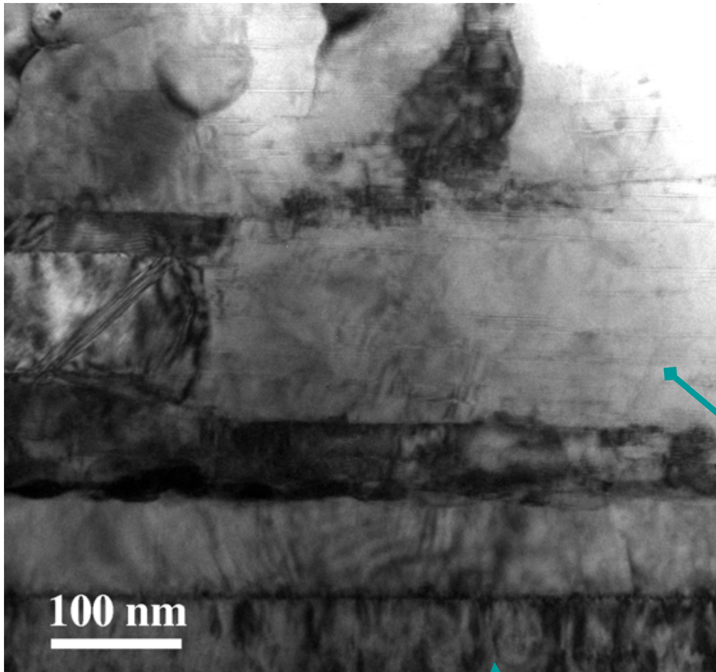


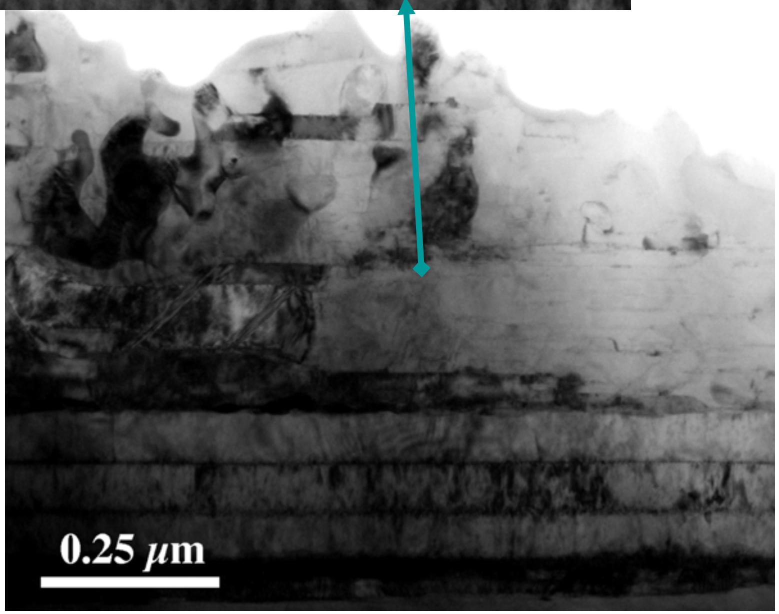


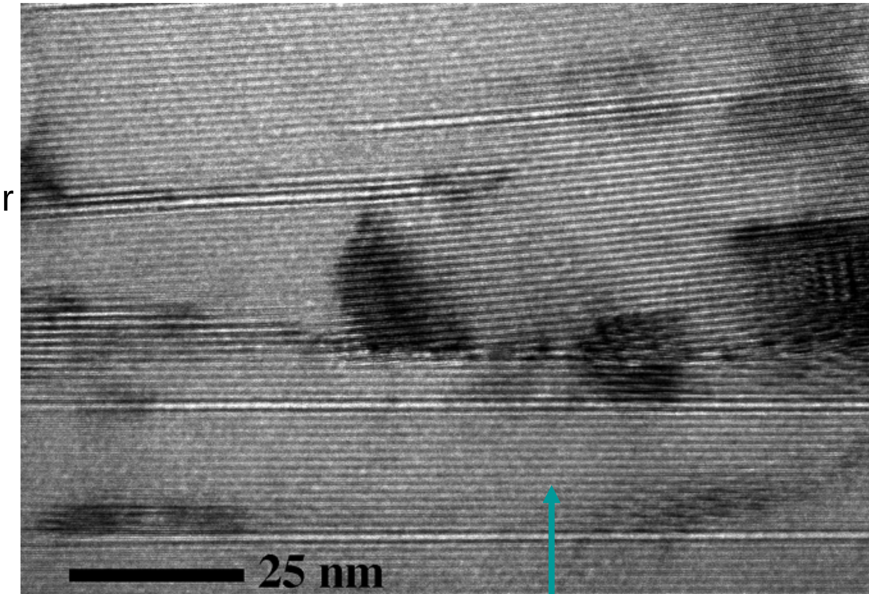

500nm

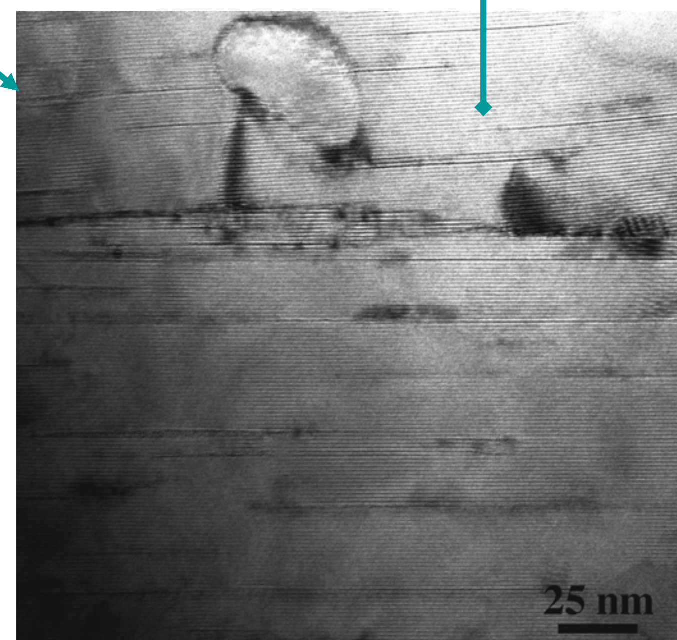
478A3

Terry Holesinger









Conclusions

- FTIR has shown signature effects in the processing of YBCO that give important information on the phase stability and phase states during formation.

- The ex-situ XRD-Dome has shown:
  - Why CeO_2 reaction with Ba is not a serious effect on the epi of YBCO.
  - Where in P, T the growth(assisted by liquid) occurs.
  - Types of growth in P, T region of stability diagram.

- Techniques developed to make MA/cm^2 on tape at 1.1um, which when etched, shown independence in thickness. This leads to a conclusion that lateral growth is the growth mode as shown by TEM.

FOUR ISSUES

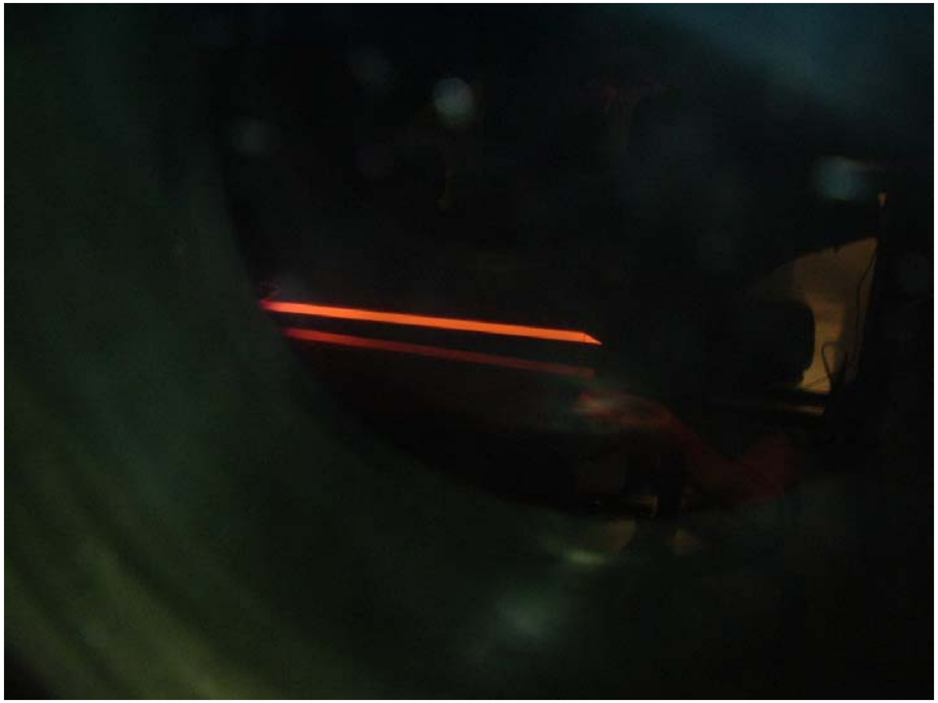
- Composition – 3 pools, different volatilities
- Oxygen – sufficient and compatible
- Temperature – controllable and uniform
- Epitaxy – compatible substrate

Reel-to-Reel Coater at LANL

- 25 kW differentially-pumped electron gun
- Tunable Laser AA (TDLAA) vapor sensors (Y, Ba)
- HCL AA vapor sensor (Cu)
- Resistively heated tape
- Specialized software
- Microwave oxygen plasma applicator

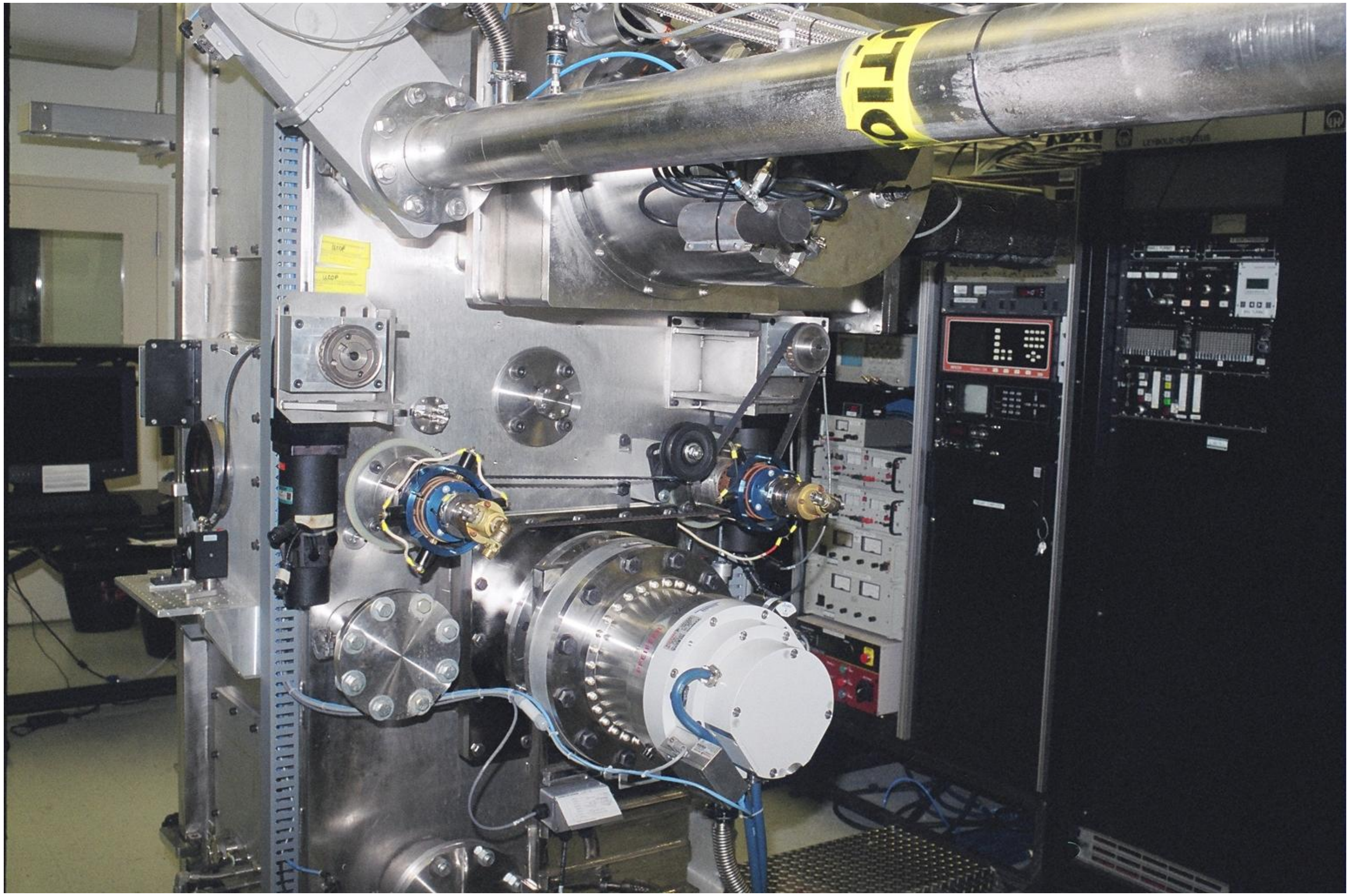
Equipment donated by 3M Company

Resistively heated tape



- Heat applied directly to tape.
- Pyrometer can view tape directly w/o hotter background.
- No burnout of quartz bulbs or plates to coat-up.
- Width & length scalable.
- Substrate thickness variations or coating conductivity can affect temperature.
- Emissivity problem same as IR heater.
- Arcing possible, conductive backside required.

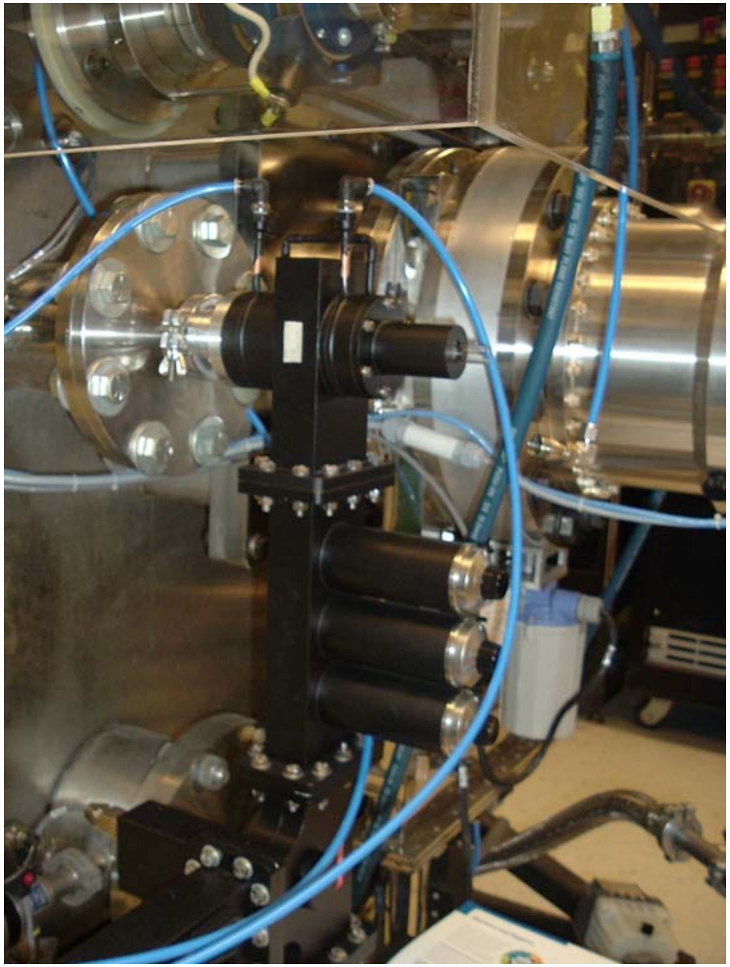
Current applied to tape through two water-cooled wheels and slip rings.



SUBSTRATES

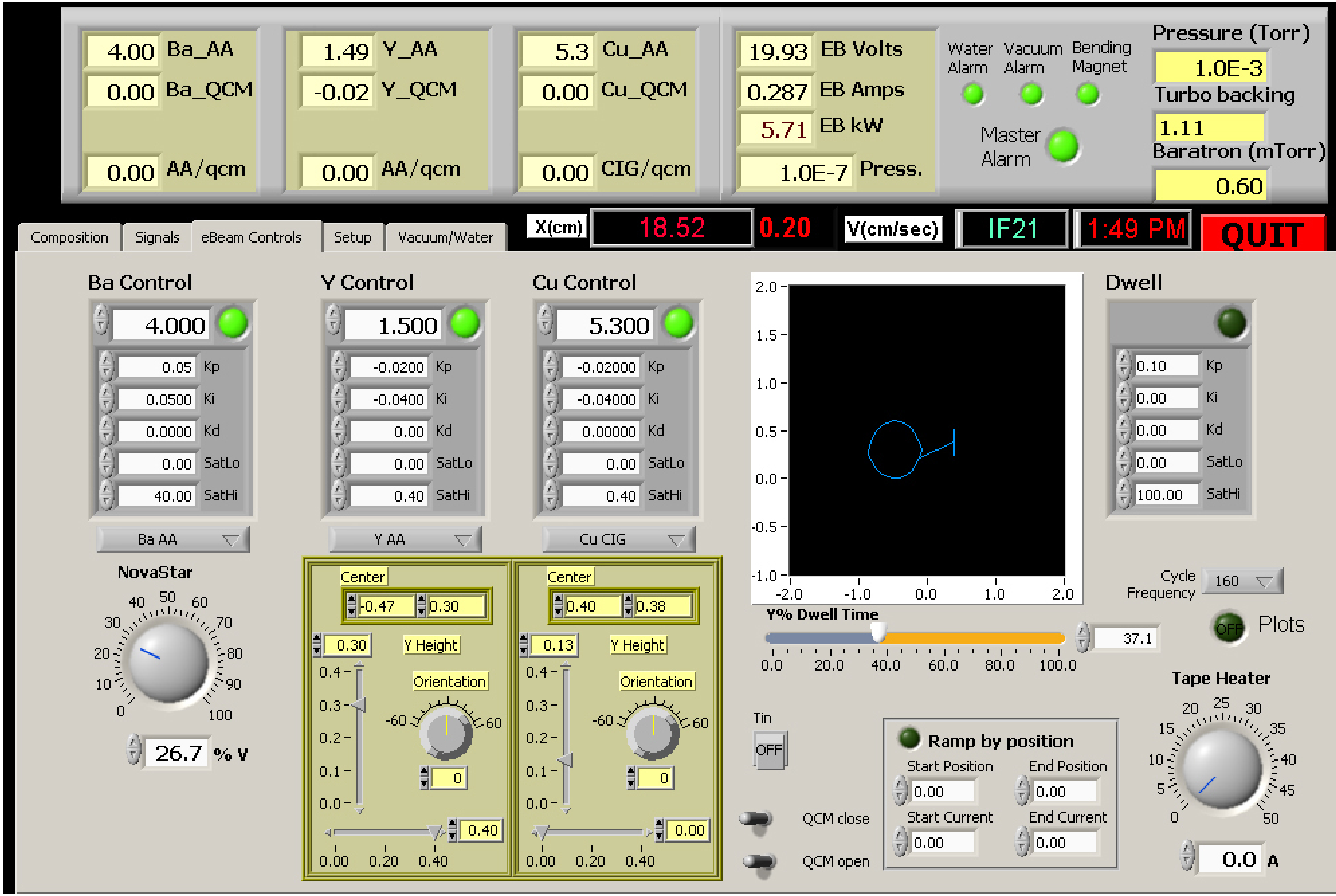
Both IBAD-MgO/CeO_2 (LANL) and RABiTS (AMSC) are being used in the LANL reel-to-reel process.

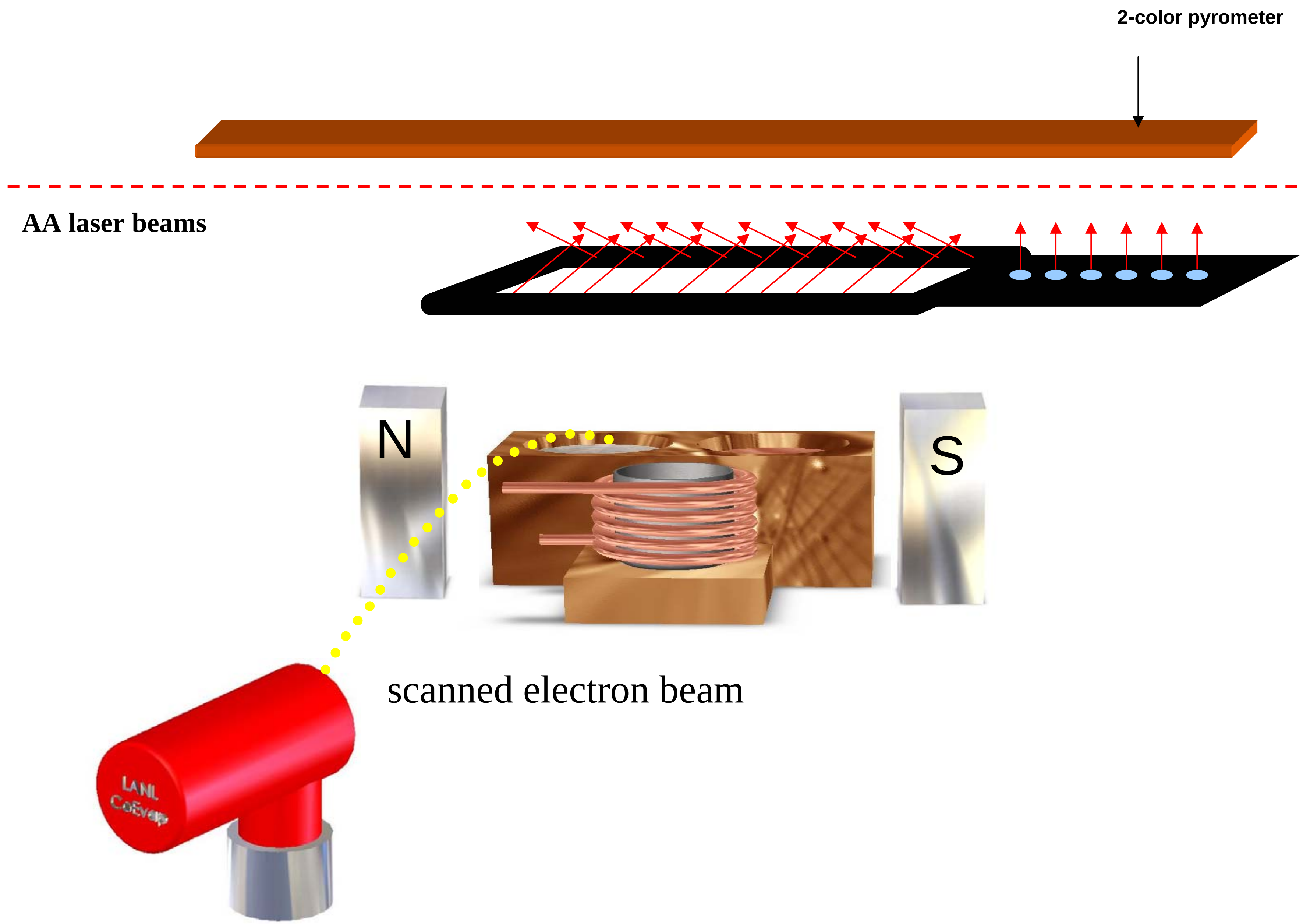
Microwave plasma to provide atomic oxygen



’06: characterize with AA and optimize

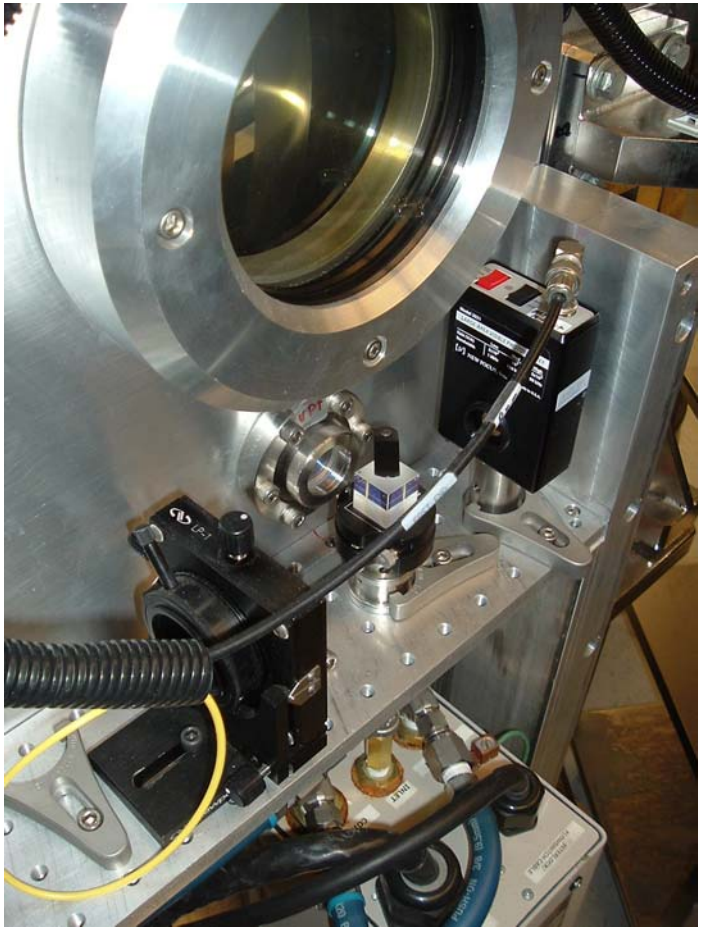
Controls for three vapor sources



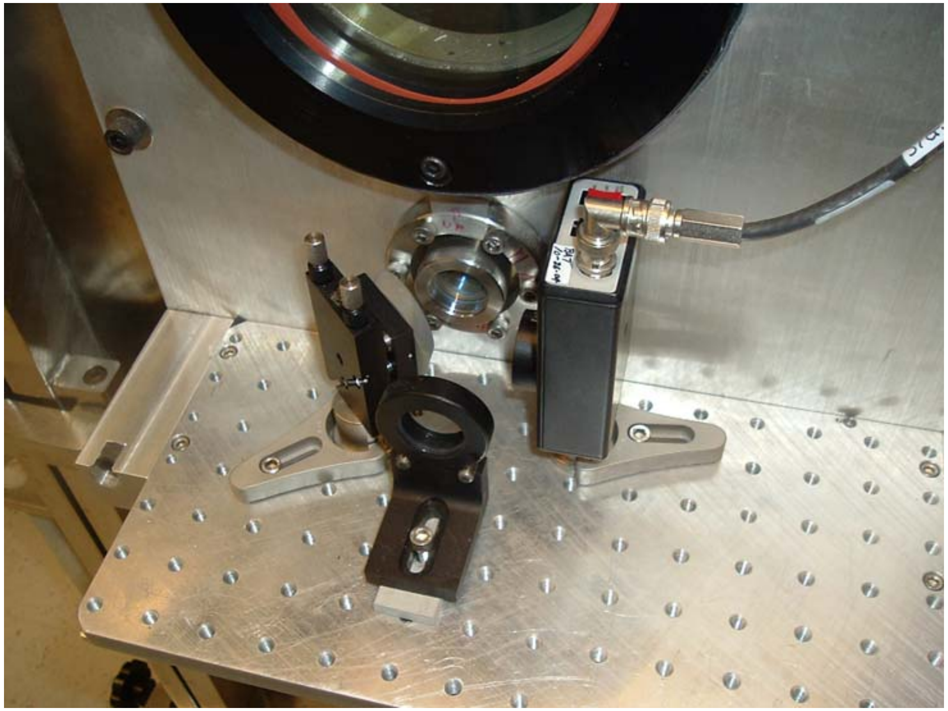

2-color pyrometer
AA laser beams
N
S
LANL
CoEvap
scanned electron beam

Atomic Absorption:
Robust, Accurate, Fast

Non-intrusive and windows do not coat-up.



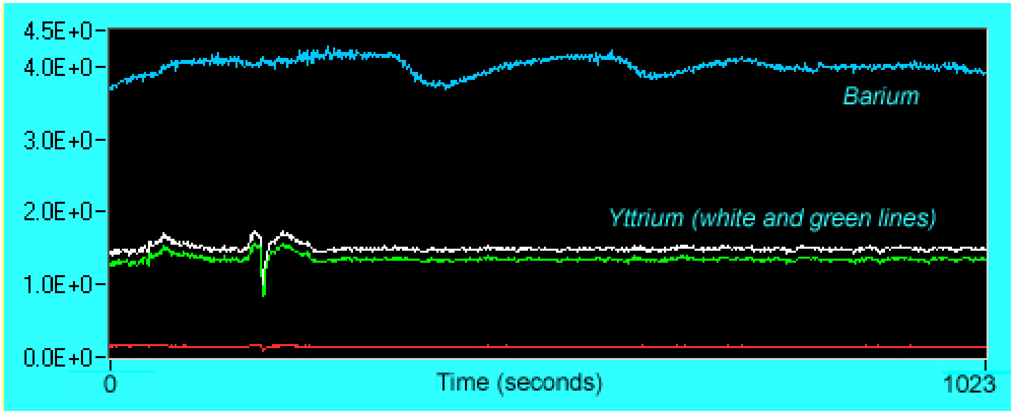
Launch

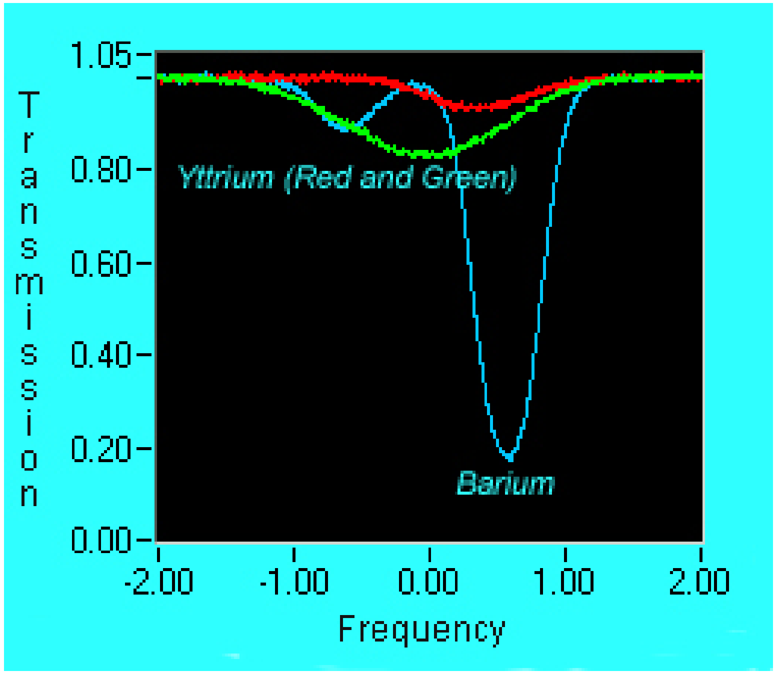


Receive

Tunable Diode Laser AA: yttrium and barium

Vapor rates

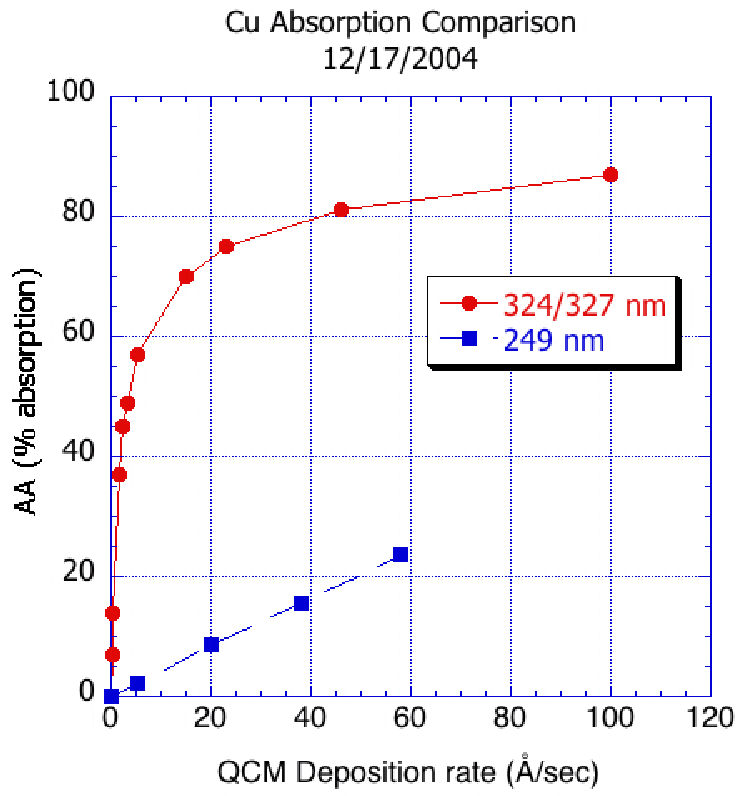


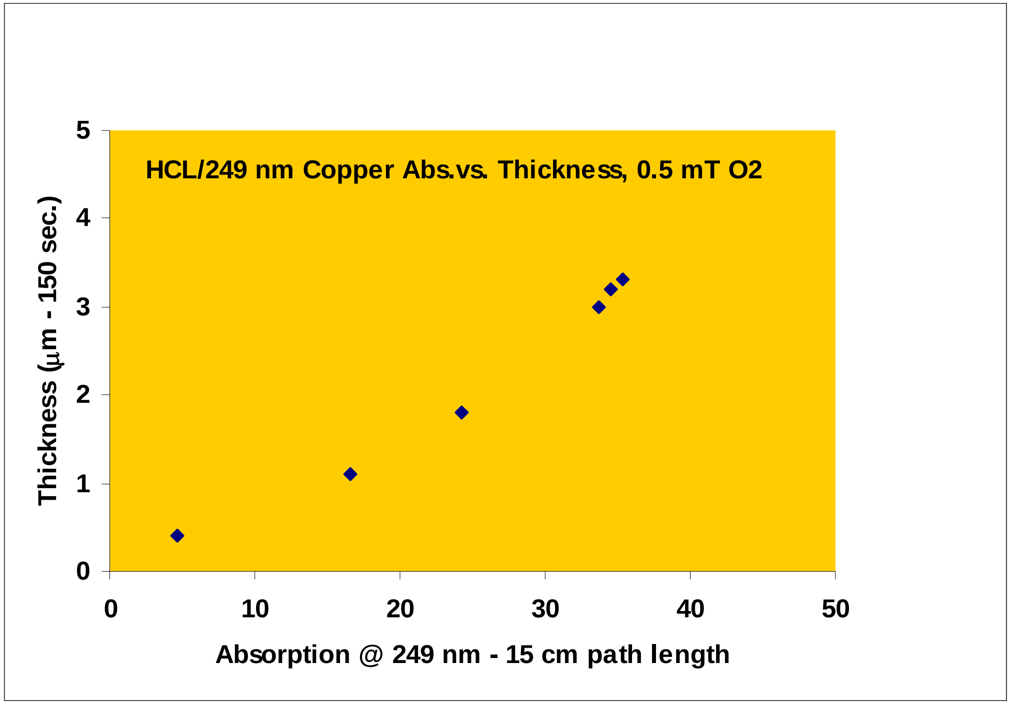


Note: ^{137}Ba 11% ^{138}Ba 72%

Atomic absorption sensor - copper

Lasers cannot reach the excessively strong ground state absorption line at 325 nm so use hollow cathode lamp (HCL)

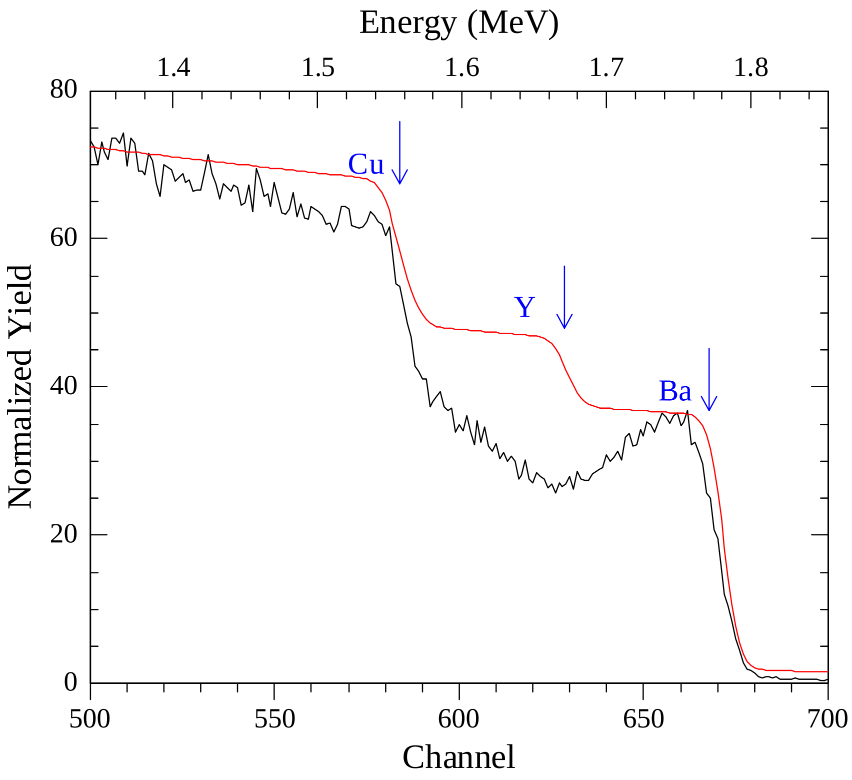




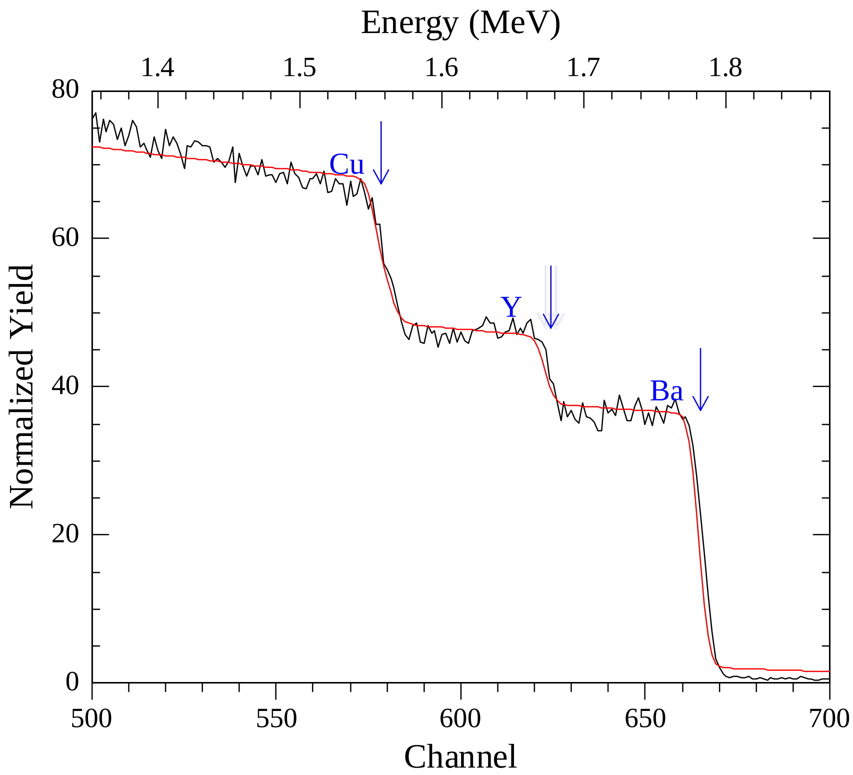
Laser for copper is still a possibility!

RBS indicated non-uniform depth distribution of elements

Non-uniform (Linear Source Geometry) → **Uniform (Triangular Source Geometry)**



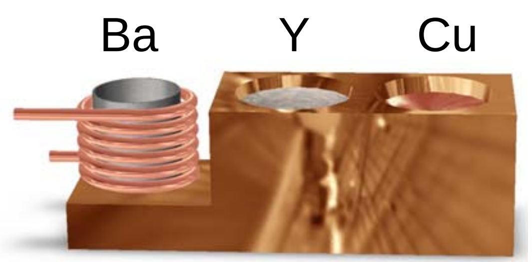
Ce036b (ICP: 17.8%, 27.0%, 55.2%)



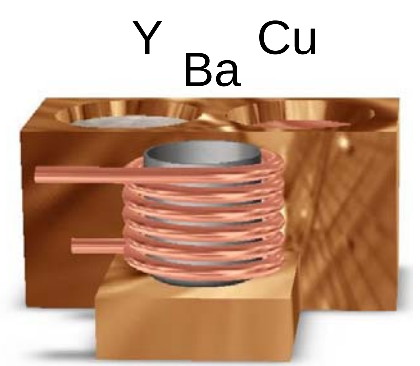
Ce046c (ICP: 20.9 at%, 26.5 at%, 52.6 at%)
(RBS : 15.4 at%, 28.6 at%, 56.0 at%)

Source Geometry Modified

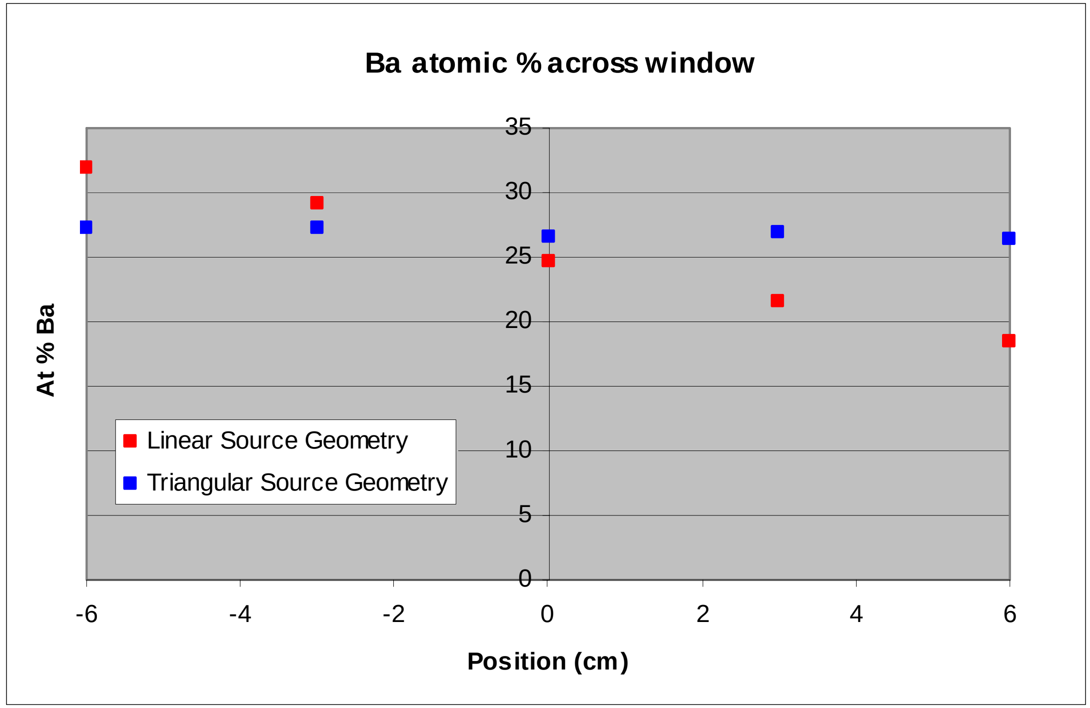
Programmable sweep Pierce-type electron gun also offers flexibility in source geometry.



Linear Source Geometry

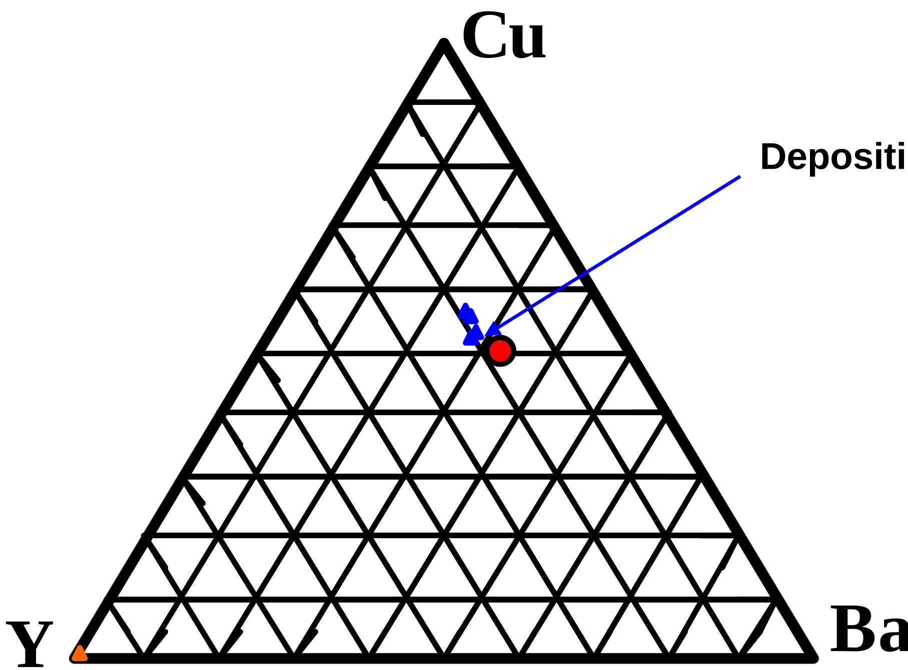


Triangular Source Geometry

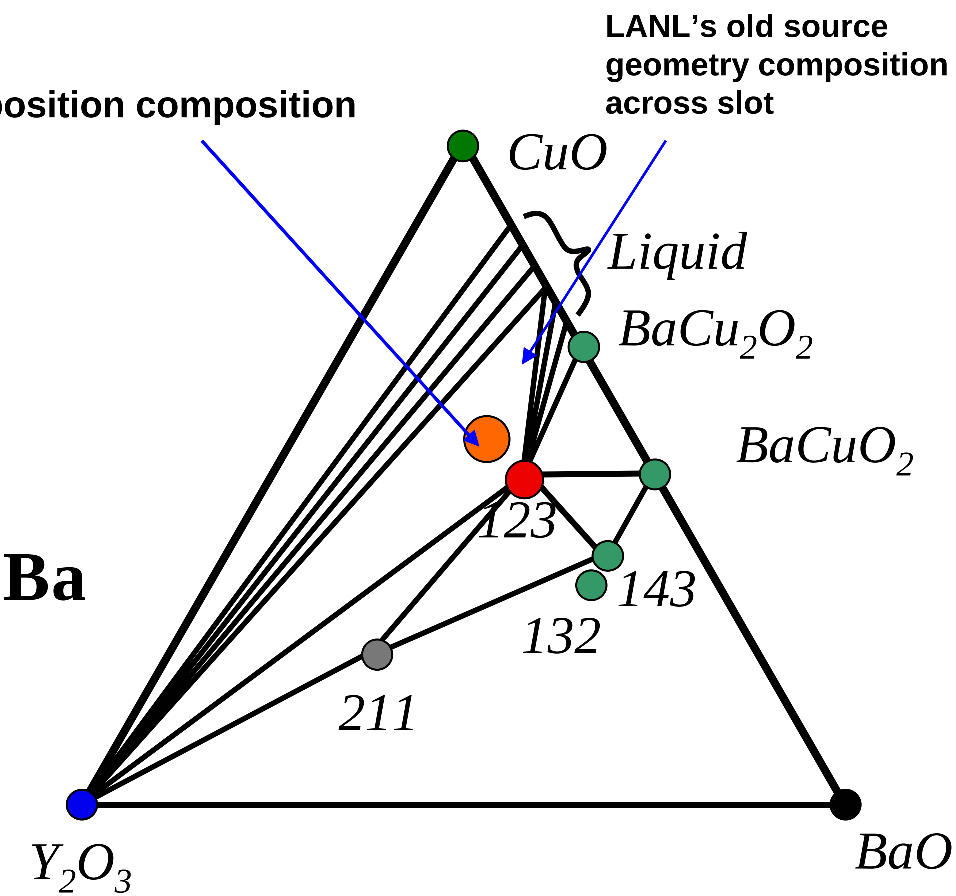


LANL Co-Evaporation Composition in agreement with Stanford research

This composition maintained > ½ hour on moving tape

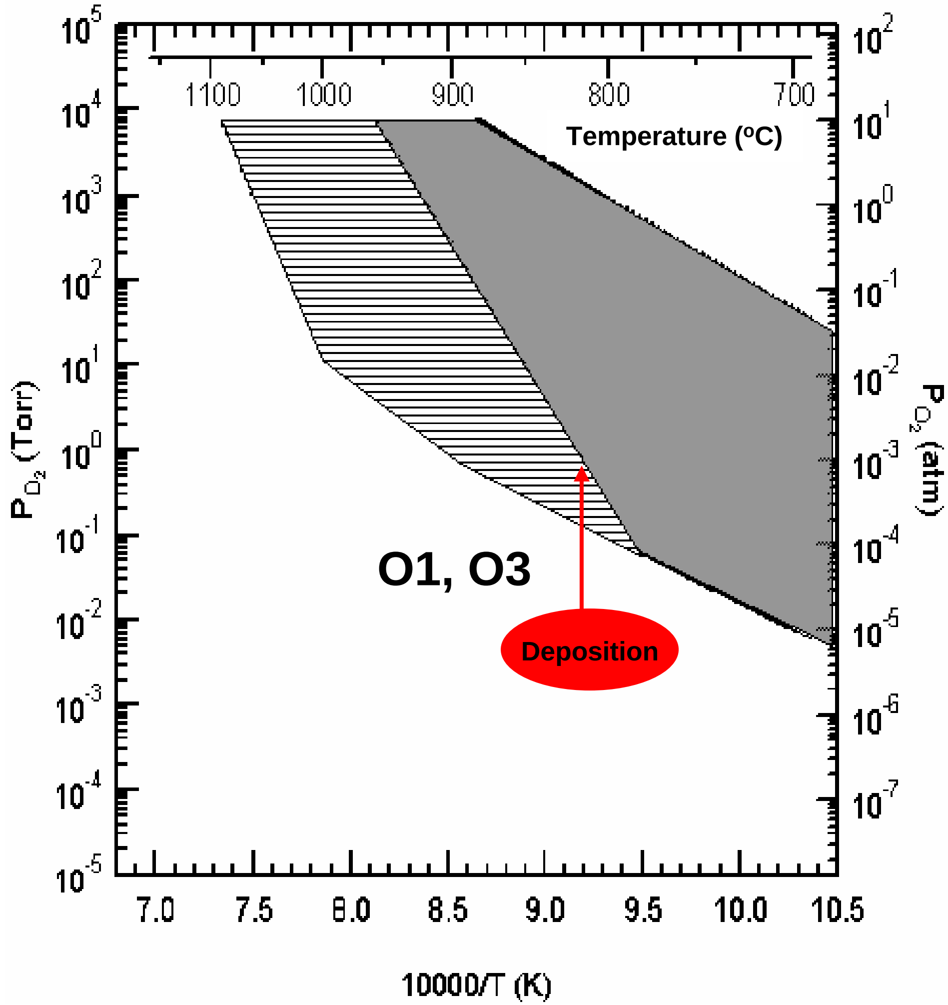


Demonstrated composition control ~ +/-2 at%



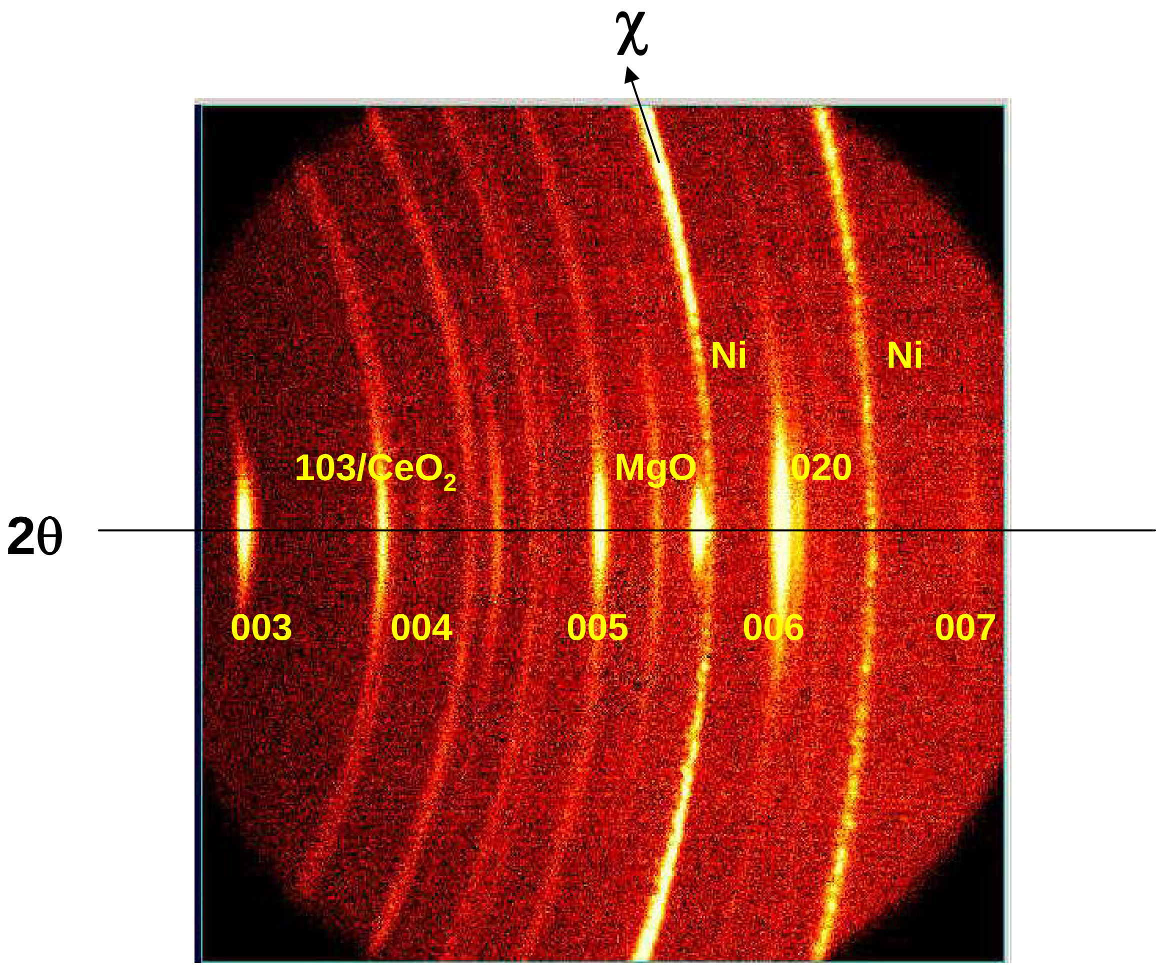
Ex situ composition determined by Inductively Coupled Plasma Emission Spectroscopy (ICP)

in situ deposition using O_1 or O_3.



Max P_{cham}=10 mT ?

YBCO on IBAD-MgO/CeO_2 has good texture



Bruker GADDS XRD system

ce056c made at LANL, annealed at Stanford

Criterion: Results

1. J_c of 1 MA/cm^2 1.1 micron thick YBCO on RABiTS.

2. LANL has an operational, tape-based, computer-driven, co-evaporation system.

3. The FTIR tool has elucidated the details of the YBCO formation.

4. The XRD-Dome tool has elucidated details of the crystallization process.

5. The moving tape co-evaporation process has produced YBCO films on RABiTS and IBAD substrates.

6. Composition control at LANL has been demonstrated.

7. A Cu AA sensor was developed.

Criterion: Performance

- ✓ **Goal: Install and improve equipment for electron beam co-evaporation at LANL.**
- ✓ **Goal: Operate equipment for electron beam co-evaporation with AA sensors, computer control and specialized software.**
- ✓ **Goal: Develop a evaporation (e-beam and thermal) deposition method capable of producing high Jc YBCO films.**
- ✓ **Goal: Produce YBCO films on RABiTS and IBAD.**
- ✓ **Goal: Develop a new atomic absorption (AA) tool for copper and demonstrate composition control (+/-2 at%) using computers for automation.**

Criterion: Research Integration

1) There is a fruitful collaboration between Stanford and LANL that has improved the total program capabilities. Stanford is mapping out the pressure-temperature stability plot which is being used by LANL to demonstrate the process on moving tape.
2) LANL, through its CRADA with American Superconductor, has obtained RABiTS. It has been used by Stanford and LANL.
3) Both partners are also using LANL IBAD substrate.
4) The LANL co-evaporation program is co-located with LANL's PLD YBCO and IBAD operations. These experts are intimately involved with this program.
5) University of Wisconsin (Dr. Matt Feldman) made measurements of Jc on RABITS.
6) LANL's TEM is being used to elucidate growth patterns.
7) LANL's RBS has shown way to uniformity.
8) XRD and electrical measurements on Stanford samples is ongoing.
9) Patent filed.

Criterion: 2006 Plans

1) Produce 1 meter of 1 cm tape with an Ic of 100 Amperes (SF, 75K).
2) Investigate post-annealing capitalizing on materials science work from Stanford. If promising, add annealing to the tape path.
3) Investigate oxygenation methods:
   a) Activated oxygen using the microwave-powered oxygen plasma or ozone.
   b) Explore higher ambient O_x pressure ~10 mT.
   c) Install and operate an "oxygen pocket" device for moving tape.
   d) Characterize and maximize O_1 generation using AA.
4) Improve the tape thermometry through pyrometer improvements and calibration.
5) Stanford and LANL conduct sample exchange, visits and information in order to achieve these plans.